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TECHNICAL REPORT FRL-TR-25

STANDARD LABORATORY PROCEDURES
FOR SENSITIVITY, BRISANCE,
AND STABILITY OF EXPLOSIVES

ARTHUR J. CLEAR

JANUARY 1961

FELTMAN RESEARCH LABORATORIES
PICATINNY ARSENAL
DOVER, N. J.

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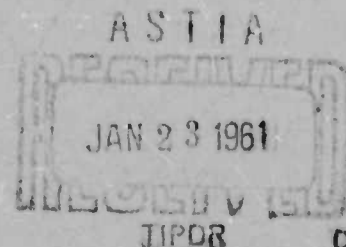
STANDARD LABORATORY PROCEDURES FOR SENSITIVITY, BRISANCE, AND STABILITY OF EXPLOSIVES

by

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January 1961

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Picatinny Arsenal
Dover, N.J.

Technical Report FRL-TR-25   Approved:

L H Eriksen

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                                              Chief, Explosives and
Dept of the Army Project: None   Propellants Laboratory

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OBJECT

To revise the directions given in Picatinny Arsenal Technical Report 1401 Rev. 1, which cover all standard laboratory tests used at Picatinny Arsenal to determine the sensitivity, brisance, and stability of explosives. The purposes of this revision are to

1. Incorporate changes which have become essential during the past ten years,

2. Eliminate procedures or tests which are considered obsolete or others which are used very infrequently, and

3. Add drawings which are in current use, and photographs which are up to date.

SUMMARY

The detailed procedures given in Picatinny Arsenal Technical Report No. 1401, Rev. 1 for standard laboratory tests to determine the sensitivity, brisance, and stability of high explosives, primers, pyrotechnic compositions, and propellants have been reviewed and revised to bring the methods up to date. Accordingly the directions for several of the procedures have been modified wherever necessary to include details which have been found to be important. Procedures which are either obsolete or seldom used have been omitted.

INTRODUCTION

Picatinny Arsenal Technical Report 1401, Rev. 1, "Standard Procedures for Sensitivity, Brisance, and Stability of Explosives," was issued in February 1950. Since that time several of the methods have been modified, replaced, or even deleted. It was considered desirable, therefore, to issue an up-to-date manual containing the standard procedures in current use at this Arsenal. Although this report is primarily intended for use as a standard within this Arsenal, other installations dealing with explosives may find some of the methods applicable to their work. For this reason, it is being given wide distribution.

1. SENSITIVITY TESTS

There follow detailed procedures for making impact tests to determine sensitivity to shock, the explosion temperature test to determine sensitivity to heat, and the sand test to determine sensitivity to initiation.

A. Impact Test, with Picatinny Arsenal Apparatus

Apparatus

The apparatus, shown on Picatinny Arsenal Drawing No. DP-2905 and in Figures 1 and 2 (pp 2 and 3) of this report, consists essentially of an anvil, two guide bars equipped with an adjustable support for a weight, and a

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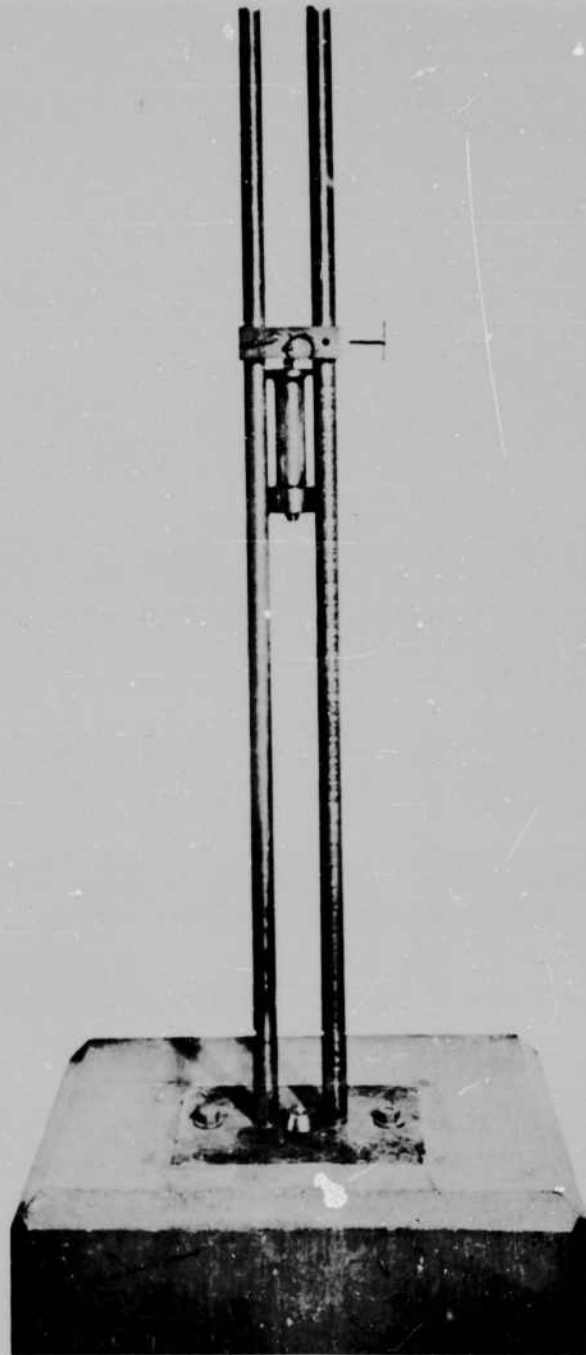
Fig 1   Picatinny Arsenal impact test apparatus

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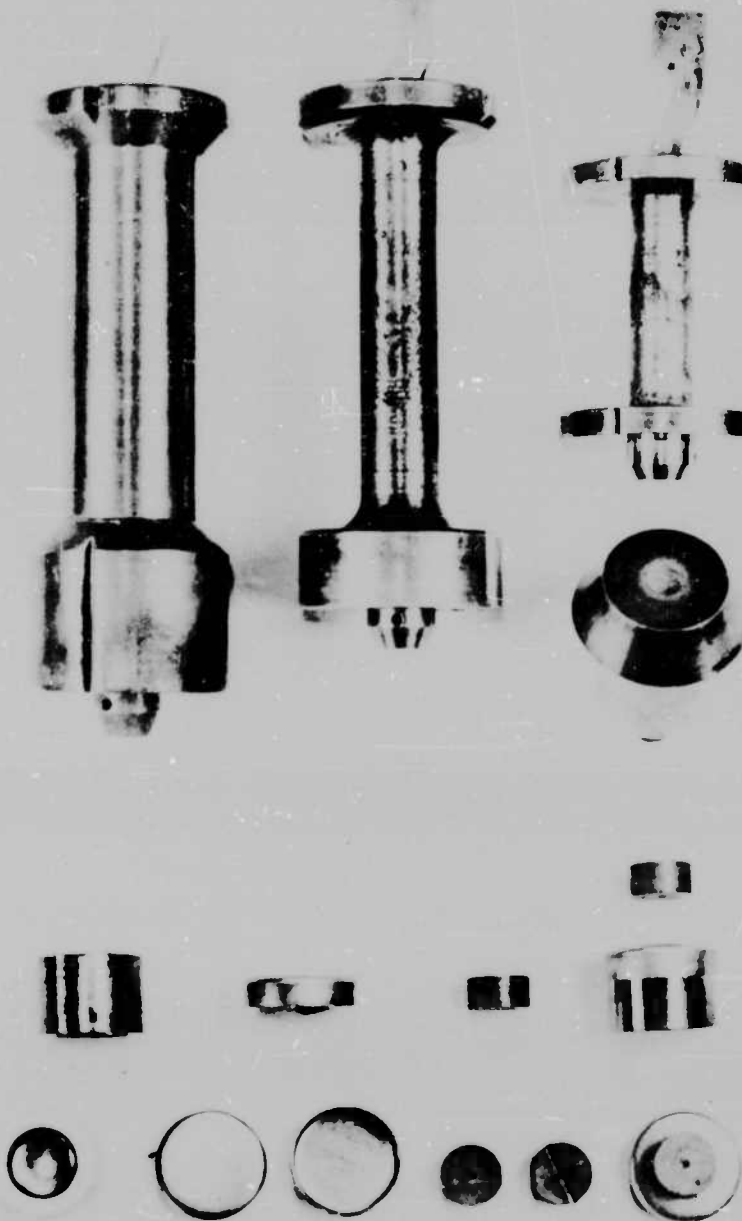
Fig 2   Weights, anvil, die cup, brass cap, and vented plug used in Picatinny Arsenal impact test

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supply of vented plugs, die cups, and die cup covers. The details of these parts are given in the following Picatinny Arsenal drawings:

DP 2905, Detailed print
BP 2906, Upright (with scale)
BP 2907, Upright
BP 2908, Scale
AP 2909, Drop weight, 2 kg
AP 2910, Indicator stop
AP 2911, Foot plate
AP 2912, Square cap
AP 2913, Anvil
AP 2914, Yoke
AP 2915, Scale clamp
AP 2916, Scale clip
AP 2919, Lock ring
AP 2920, Lock guide
AP 2921, Stud-upright
AP 2922, Hammer lock
AP 2923, Lock spring
AP 2924, Vented plug
AP 2925, Brass cap
AP 2926, Die cup
AP 2927, Regulating pin
AP 2933, Drop weight, 1 lb
AP 2934, Drop weight, 1 kg
AP 9937, Yoke stop screw
AP 9938, Yoke stop screw pin
AP 9967, Yoke
AP 9968, Suspension hook
AP 9969, Suspension hook pin
AP 9970, Anvil

The steel anvil, which has a recessed opening to support the die cup, is supported by a steel sheet which in turn is imbedded in a large block of reinforced concrete. Two brass bars, approximately 6 feet long, are securely fastened to the steel plate at opposite sides of the anvil. They extend up perpendicularly from the plate to a rigid support which is attached to a wall or some other suitable support. The insides of these bars have tongues which serve as guides for a falling weight. A yoke equipped with a release pin is attached to the two bars so that it can be moved to any desired height and held in place by hand screws. This impact test machine is used with weights of 2 kilograms, 1 kilogram, or 1 pound. The lighter weights are for use with the more sensitive materials, such as lead azide. The weight selected is inserted between the parallel bars so that the tongues of the bars fit into the grooves of the weight. The weight is fastened to the yoke by means of a pin support.

The die cups are made of hardened tool steel and the vented plugs are constructed of high-carbon drill rod drawn and hardened. The hardness values of the die cups, weight tip, base block, and vented plugs must be in accordance with the requirements of the applicable drawings. In addition, carefully prepared samples of standard military high explosives (of specification grade) must give the following test values:

Explosive	Impact Test with 2 kg Weight, in.
Tetryl	7 to 8
TNT	14 to 15

The covers used on the die-cup assembly are 70/30 brass having a Rockwell hardness of 70 to 85 on the B scale.

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Procedure

For this test, use only explosives of such granulation that they pass through a No. 50 U.S. standard sieve and are retained on a No. 100 U.S. standard sieve. Fill 10 previously weighed die cups with an excess of the explosive being tested and strike off the excess by means of a plastic or wooden spatula. Remove any explosive from the rim of the cup. Weigh the 10 loaded die cups and record the average weight of the explosive in each cup. This will give a measure of the charge density of the explosive under test. The charge density may vary depending on the crystal form, shape, and size distribution.

Load in a similar manner a sufficient number of charges for the test (at least 30 assemblers will be needed). Place a brass cover on each loaded die cup and press it down by means of a small arbor press so that the cover is in contact with top rim of the die cup. The arbor press used at Picatinny Arsenal is shown in Figure 3 (p 6). The ram of the press is barricaded with a cylindrical, plastic shield.

Place a die cup assembly in the anvil with the brass cover upward. Place a vented plug on top and in the exact center of the brass cover, with the grooved side of the plug downward. Adjust the yoke so that the weight will fall about 12 inches for the first trial. If explosion[1] occurs, lower the hammer

about 2 inches for the next trial and continue the adjustment for different trials until the desired height of drop, as explained below, is reached. If no explosion is obtained on the first trial at 12 inches, raise the weight about 4 inches for the next trial and so on until the desired height of drop is found. The purpose of these preliminary tests is to determine the approximate drop test value for the explosive, and until such a value is obtained it is not necessary to make more than one trial at each height.

Use a new cup or one with a smooth inner surface, a new vented plug, and a new brass cover for each trial. The assemblies may be disassembled after test by using the fixture shown in Figure 4 (p 7). The explosive may be removed from the die cup by means of a suitable solvent. Record as the impact test value the minimum height in inches from which the impact of the falling weight causes explosion of the sample in at least one of ten trials, and from one inch below which the impact of the falling weight causes no explosion in 10 trials.

Note: This apparatus can be used for determining the sensitivity of the very sensitive as well as the least sensitive explosives, and is particularly suitable for testing the least sensitive military explosives such as TNT and ammonium picrate.

B. Impact Test, with U.S. Bureau of Mines Apparatus

Apparatus

The apparatus for this test, shown in Figures 5 and 6 (pp 8 and 9), consists

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[1] In this test, an explosion in defined as any audible or visual evidence of decomposition such as a crack, flash, smoke, or charring.
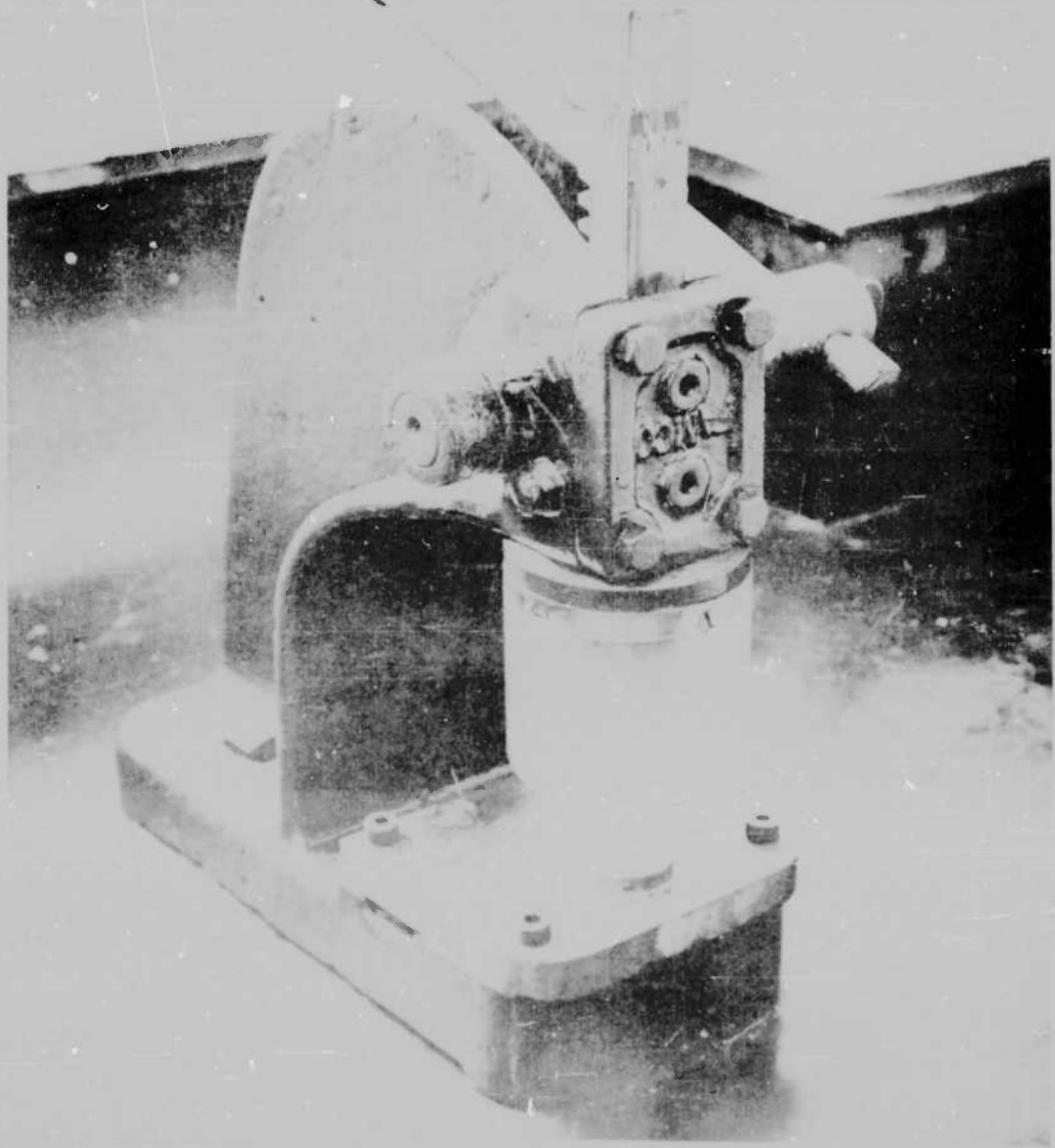
Fig. 5   Arbor press for assembling brass caps on the cups used in Picatinny Arsenal impact test.
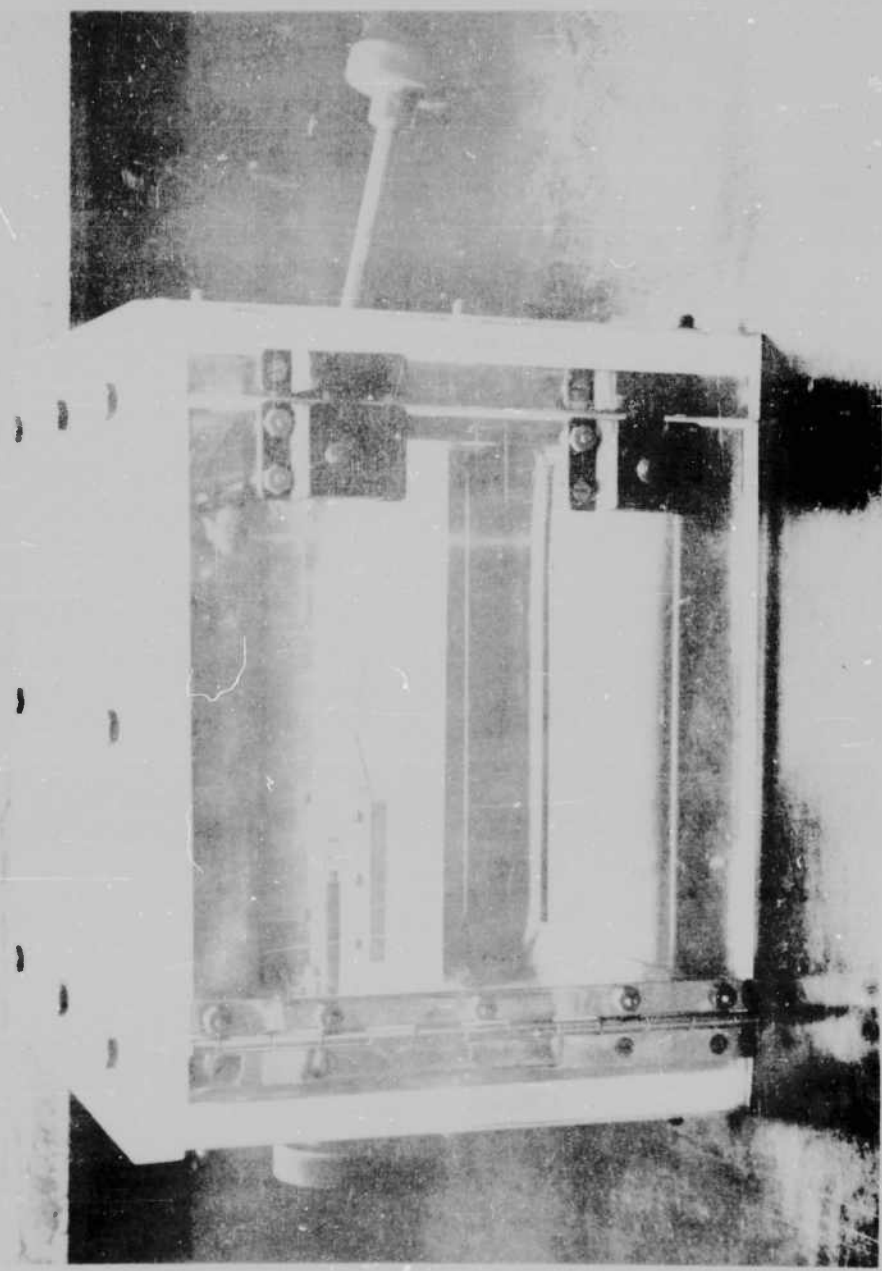
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Fig. 4   Fixture used for disassembling brass cover from die cup used in Picatinny Arsenal impact test
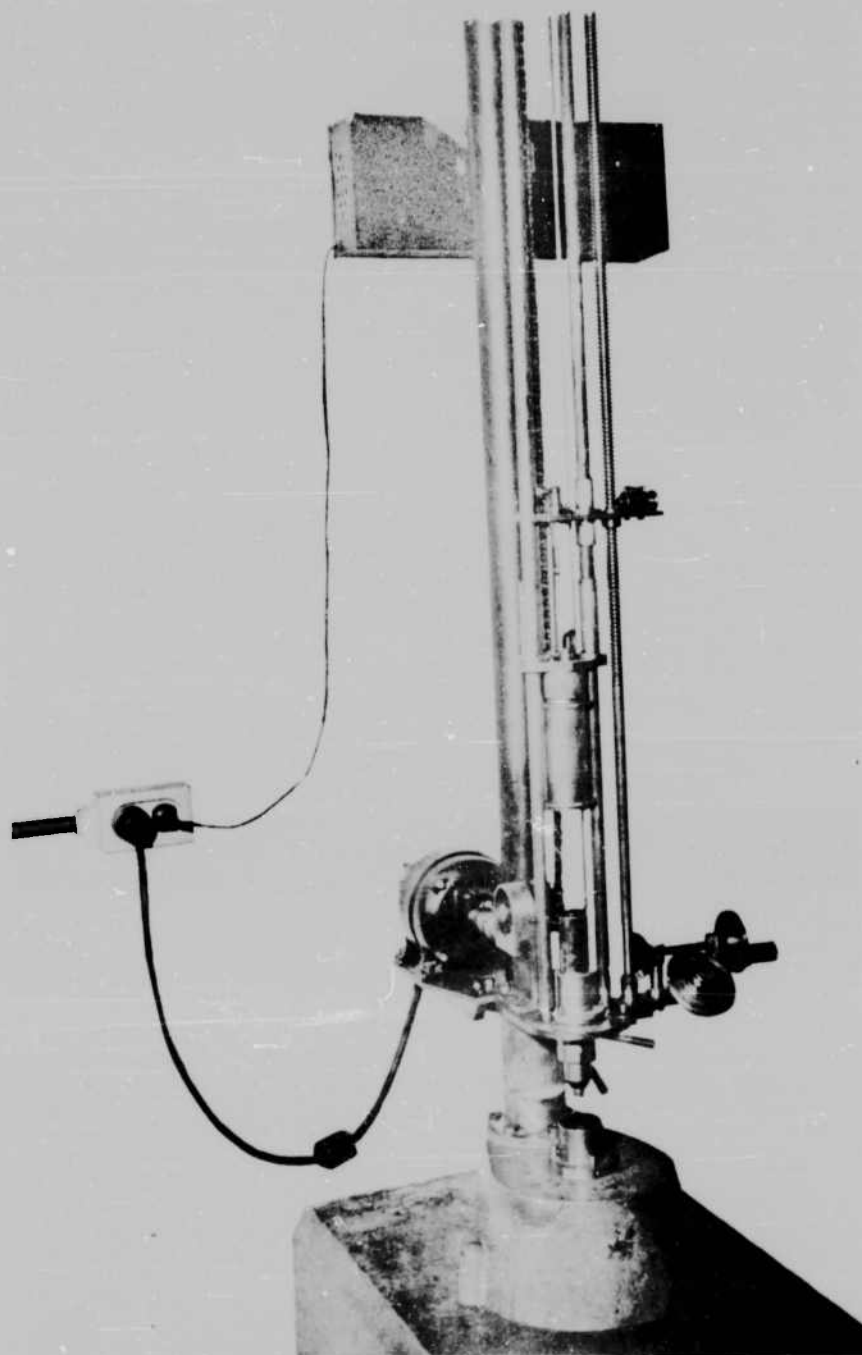
Fig 5   Bureau of Mines impact test apparatus

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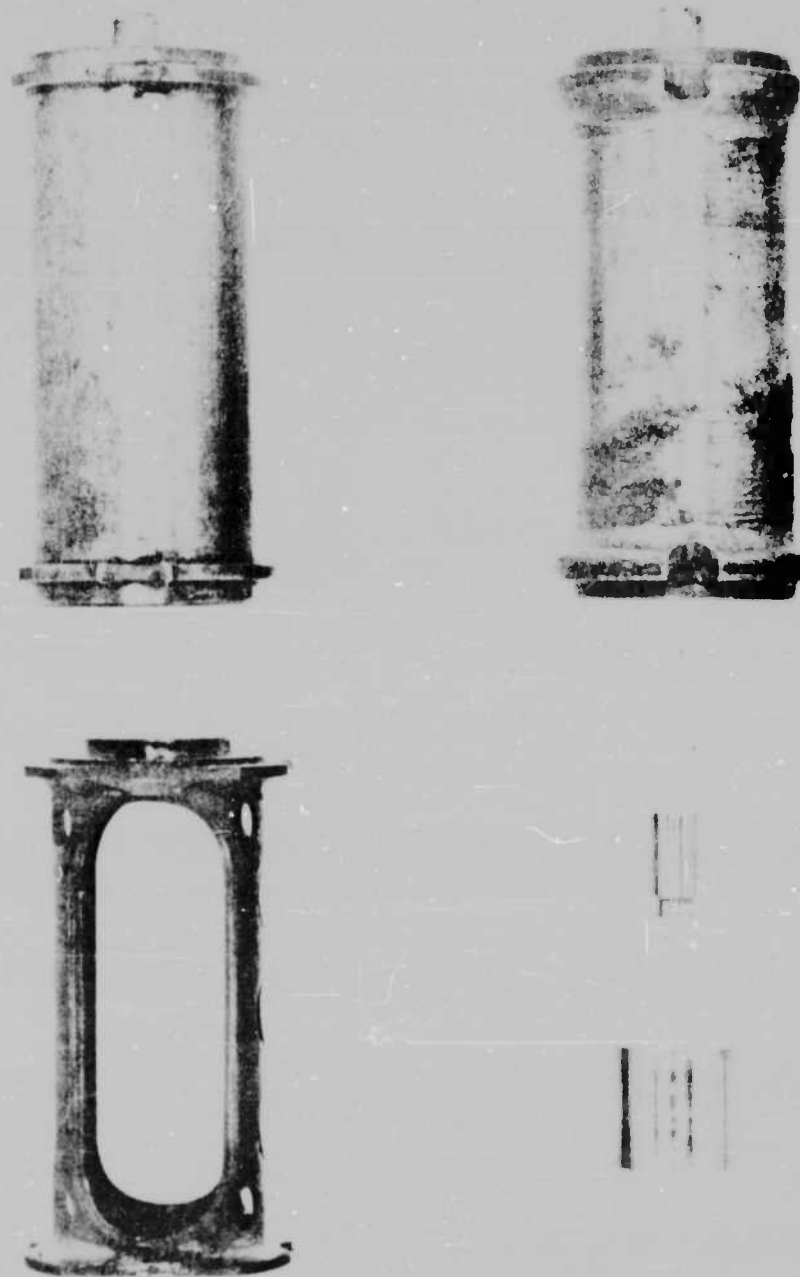
Fig 6   Weights, plunger tip, and base block for Bureau of Mines impact test apparatus

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essentially of an impact block, a plunger, and a weight which can be dropped from any desired height from 1 to 100 centimeters. Detailed dimensions of the base block and plunger tip are given in Figure 7 (p 11).

The impact block has an easily replaceable insert of hardened steel having a Rockwell hardness of C-63 ± 2, and is strongly supported by a metal base imbedded in a large block of reinforced concrete. The hardened steel insert has a very uniform, smooth, mirror-like surface (2μ inch, rms).

The plunger is located directly over the impact block and is supported by a heavy framework which is imbedded in the same base as the impact block. The plunger is equipped with a replaceable hardened steel tip 1 cm in diameter -having a Rockwell hardness of C-63 ± 2. The flat end of the plunger tip has a mirror-like surface similar to that of the impact block. The plunger is held in position by a metal sleeve which fits snugly, and the apparatus is so constructed that the plunger can be raised about 2.5 centimeters above the block and locked in this position by means of a rod which extends out horizontally from the plunger.

Three weights of 500, 1000, and 2000 grams are used with the apparatus. These weights can be used only one at a time. The weight is inserted between two guide bars extending from the plunger frame to the frame at the top of the apparatus. The weight is raised to the

desired height by means of an encased magnetic coil held in place by sleeves attached to the two guide bars. A contact point on the yoke breaks the electromagnetic circuit and allows the weight to fall when it is touched by the contact point on the top of the magnetic coil. The yoke is supported by a screw lift which is in the same plane as the guide bars and parallel to them. It can be set at any desired height by means of the screw lift, which is equipped with a dial indicating the height of fall of the weight in centimeters. (The zero point on this dial should be checked before each use of the machine.) The magnetic coil assembly is raised by a motor-driven chain hoist supported by two sprockets located at the top and bottom of the guide bars. The revolving chain engages the magnetic assembly by means of a pin extending out on both sides of the chain. After the contact point on the magnetic assembly makes contact with the contact point on the yoke and allows the weight to fall, a rod with a cone-shaped tip extending down from the yoke releases the magnetic assembly from the chain hoist pin by spreading apart the spring-held supports extending out from the assembly, and allows the assembly to fall slowly down to the weight.

The impact block insert and plunger tip should be examined frequently to insure that the plunger tip rests evenly on the impact block insert so that the force of impact is distributed uniformly over the entire area between the plunger tip and impact block insert. This can be

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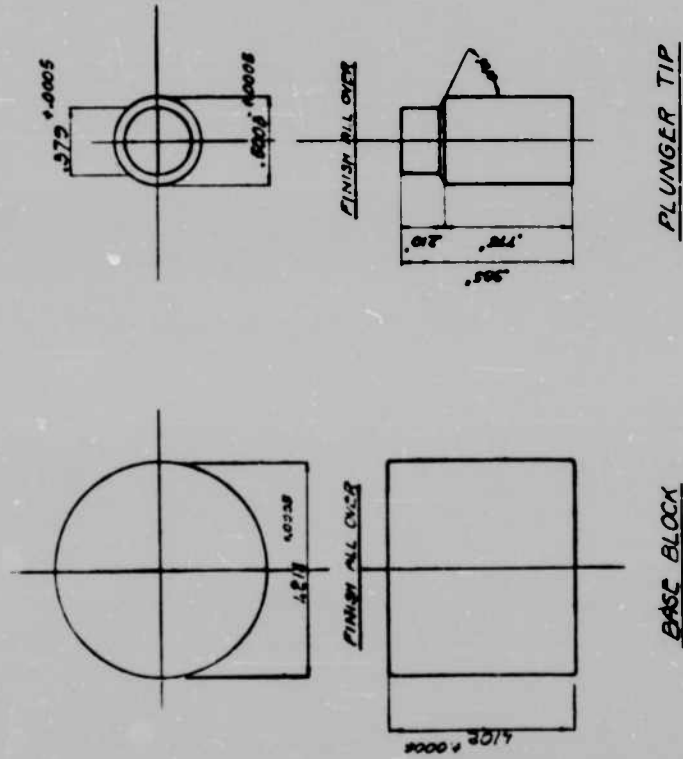
Fig 7   Details of plunger tip and base block for Bureau of Mines apparatus

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determined easily as follows: Place a piece of carbon paper with the inked side downward on top of a piece of plain white paper and place the two sheets on the impact block (with the plain paper on the bottom). Bring the plunger tip down to rest on the carbon paper and then drop the weight from a convenient height so as to make a visible imprint on the plain paper. The carbon imprint on the plain paper should be uniform in color density. If it is found that the impact block is unsatisfactory, place small pieces of thin metal sheet under the low portion until a satisfactory position is obtained.

When the apparatus is assembled it should be checked by obtaining impact test values for several standard explosives which have widely different drop test values, such as lead azide, tetryl, and TNT. The test values for standard lead azide, tetryl, and TNT are 17 cm, 26 cm and 100+ cm, respectively, when a 2-kilogram weight is used.

Procedure

Lock the plunger in the raised position and insert the weight desired between the guide bars. Close the magnetic circuit and bring the weight into contact with the magnetic lifter. Set the upper contact point so that the weight will be released from such a height that the resulting impact is expected to cause explosion. Use for this test an explosive of such granulation that it will pass through a No. 50 and be retained on a No. 100 U. S. standard sieve.

Weigh 0.020 gram of the explosive and place the weighed sample in the center of the impact block, which must be maintained at a temperature of $25° ± 2°C$. Place a shield in front of the impact block as a safety precaution. Then slowly lower the plunger on the sample and turn the plunger rod from side to side so that the layer of the sample is uniformly distributed and extends just to the edge of the plunger tip. If the sample is not visible around the entire edge of the tip, raise the plunger, lock it into place, and recenter the sample.

Start the motor which operates the chain hoist and as soon as the weight falls, shut off the motor switch. If no explosion occurs, as indicated by absence of a flash, smoke, or noise, remove the sample from the impact block and plunger tip and repeat the test. Drop the weight from a greater height in each successive trial until explosion occurs. Record as the test value the minimum drop height in centimeters at which the falling weight causes explosion of the sample in at least one of ten trials and from one centimeter below which the falling weight causes no explosions in ten trials.

After each explosion, carefully clean the block and plunger tip with fine abrasive paper. (A carborundum or emery abrasive paper of grit No. 0 has been found satisfactory.) When cleaning the tip, place the carborundum paper on the block and allow the tip and plunger to rest on the paper. Then move the paper

horizontally between the block and plunger tip so that the surface of the tip does not become rounded at the edges. If either the impact block or the plunger tip is dented, cracked, or deformed in any way, replace it before making further tests.

C. Modified Impact Test for Liquid Explosives (Made with Bureau of Mines Apparatus)

Procedure

The procedure given above under "B. Impact Test made with U. S. Bureau of Mines Apparatus" should be followed with the following exceptions: Use 0.007 ± 0.002 gram of explosive for each trial instead of 0.020 gram. Transfer a drop of the explosive to the center of the anvil and place a disc of dried filter paper, Whatman No. 1 or equivalent, 9.5 mm in diameter on top of the liquid. (The paper should be dried for an hour at $95°$ to $100°C$ and stored in a small desiccator containing anhydrous calcium chloride.) Instead of turning the plunger rod from side to side as prescribed for solid explosives, merely lower the plunger onto the disc making sure that the disc is centered under the plunger tip (Ref 1).

D. Modified Impact Test for Liquid Explosives (Made with Picatinny Arsenal Apparatus)

Procedure

The procedure given above under "A. Impact Test, with P A Apparatus

should be followed with the following exceptions: For each trial, add just enough of the liquid explosive to fill the die cup instead of adding an excess and striking off as in the case of solid explosives. Care should be taken to make sure that none of the liquid is on the rim of the die cup during the assembly of the test fixture (Ref 2).

E. Explosion Temperature Test

Apparatus

The explosion temperature test apparatus consists of a multiple-unit electric furnace equipped with a rheostat to control the temperature. A stainless steel crucible with a capacity of approximately 100 cc and a wall thickness of approximately $\frac{1}{4}$ inch is placed in the electric furnace. This crucible contains approximately 75 cc of Wood's metal, an alloy composed of 50% bismuth, 25% lead, 12.5% tin, and 12.5% cadmium which melts at about $65.5°C$. A calibrated thermocouple is immersed in the molten metal bath and connected to an indicating thermoelectric pyrometer, graduated in $10°$ intervals to cover a temperature range of $0°$ to $1400°C$. A shatterproof glass barricade, firmly supported, should be placed around and on top of the apparatus. The arrangement of the apparatus is shown in Figure 8 (p 14).

Procedure

Load ten No. 8 commercial blasting caps by placing in each 0.02

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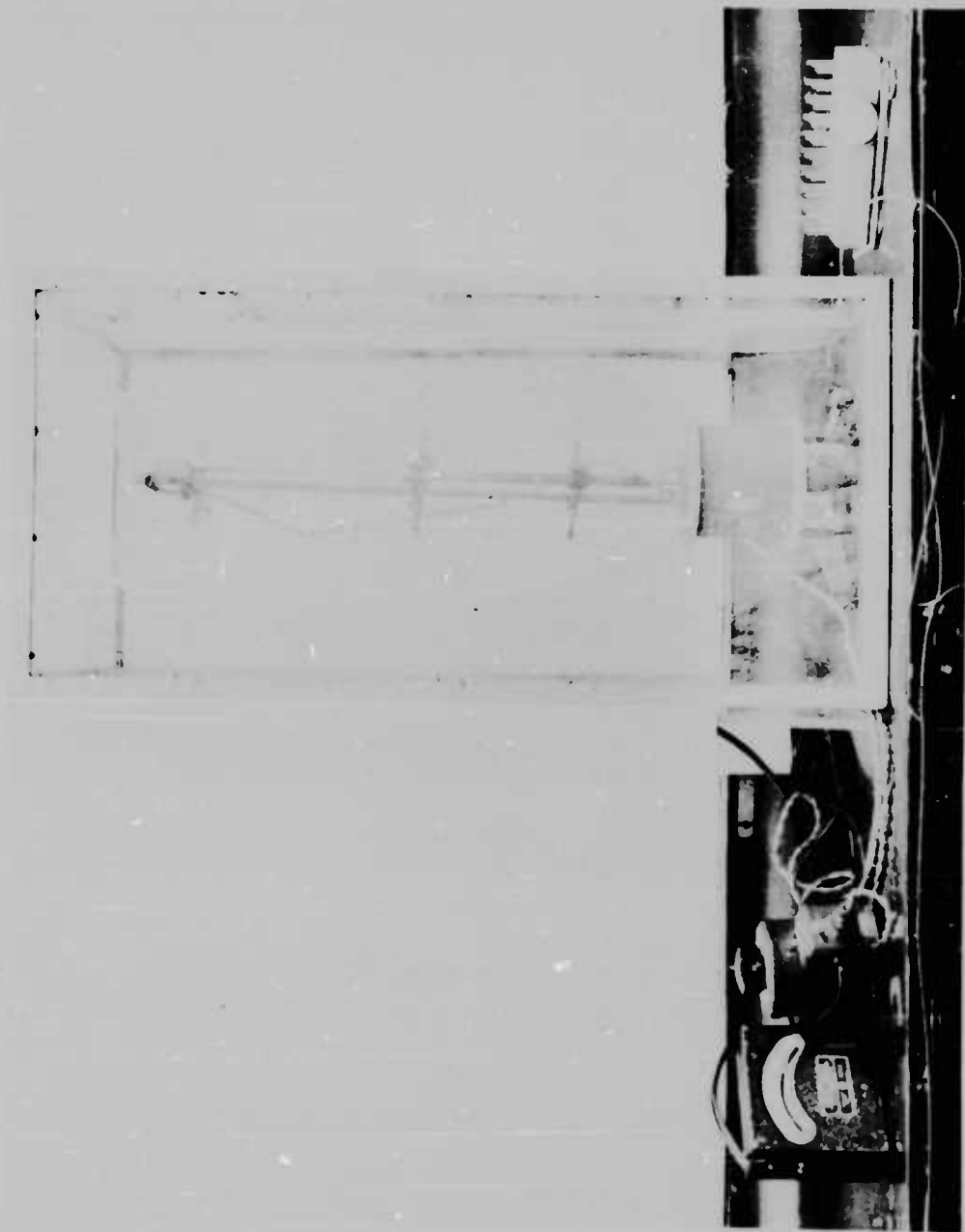
Fig. 5   Apparatus for explosion temperature test

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gram[1] of the material to be tested, of such fineness as to pass through a No. 50 U.S. standard sieve. Then tap the cap sufficiently to compact the explosive. Using insulated gloves and a pair of tongs, immerse the loaded caps in succession in the metal bath, and determine the bath temperature and the time of immersion required to cause each to flash or explode. Vary the temperature of the molten-metal bath between determinations in such a way as to have the 10 flashes or explosions occur over a time-interval range of approximately 2 to 10 seconds. Plot a time-temperature curve and select from this curve the temperature needed to cause the explosive to flash or explode after immersion in the molten bath for 5 seconds. Record this temperature as the ignition temperature of the explosive under test. With certain explosives which exhibit erratic ignitability, it is necessary to plot the average of from 2 to 5 explosions occurring over the range from 2 to 10 seconds to obtain a valid time-temperature curve.

F. Sensitivity to Initiation as Determined by Sand Test

Place the explosive in No. 6 commercial blasting caps made of copper, gilding metal, or aluminum and having an internal diameter of 0.222 – 0.002 inch. If the apparent density of the explosive to be tested is too low to permit use of a No. 6 cap, use a No. 8 cap having the same internal diameter.

[1] 0.01 gram of initiating explosives, such as lead azide.

Assemble the body of the fixture (Fig 9, p 16) to the base. Then place the empty blasting cap (open end upward) inside the body of the fixture. Assemble the funnel portion of the fixture to the top of the body.

Transfer a 0.400-gram portion of the explosive to be tested to the funnel. The explosive should be of such granulation as to pass through a No. 100 U.S. standard sieve. Insert a reinforcing cap so that the end with the greater diameter is in contact with the explosive. Transfer the fixture to a suitably barricaded loading press such as those shown in Picatinny Arsenal Drawings CP-15481, CP-23923, and SK 3857 (See Fig 10, p 17). Subject the explosive charge to a pressure of 3000 psi (111 lbs actual load) for three minutes.

Upon completion of the pressing operation, disassemble the base from the loading fixture and remove the blasting cap by allowing it to gently drop into a conductive rubber container. Pick up the loaded cap with tongs and transfer it to the crimping fixture (a Du Pont Superior Crimper obtained from the E. I. du Pont Co., Wilmington, Del., has been found convenient for this crimping operation.)

If the blasting cap does not fall free from the fixture after the pressing operation, disassemble the top funnel portion of the fixture, place the fixture in the disassembly press, close the barricade door and gently apply pressure to eject the cap into the crimping fixture.

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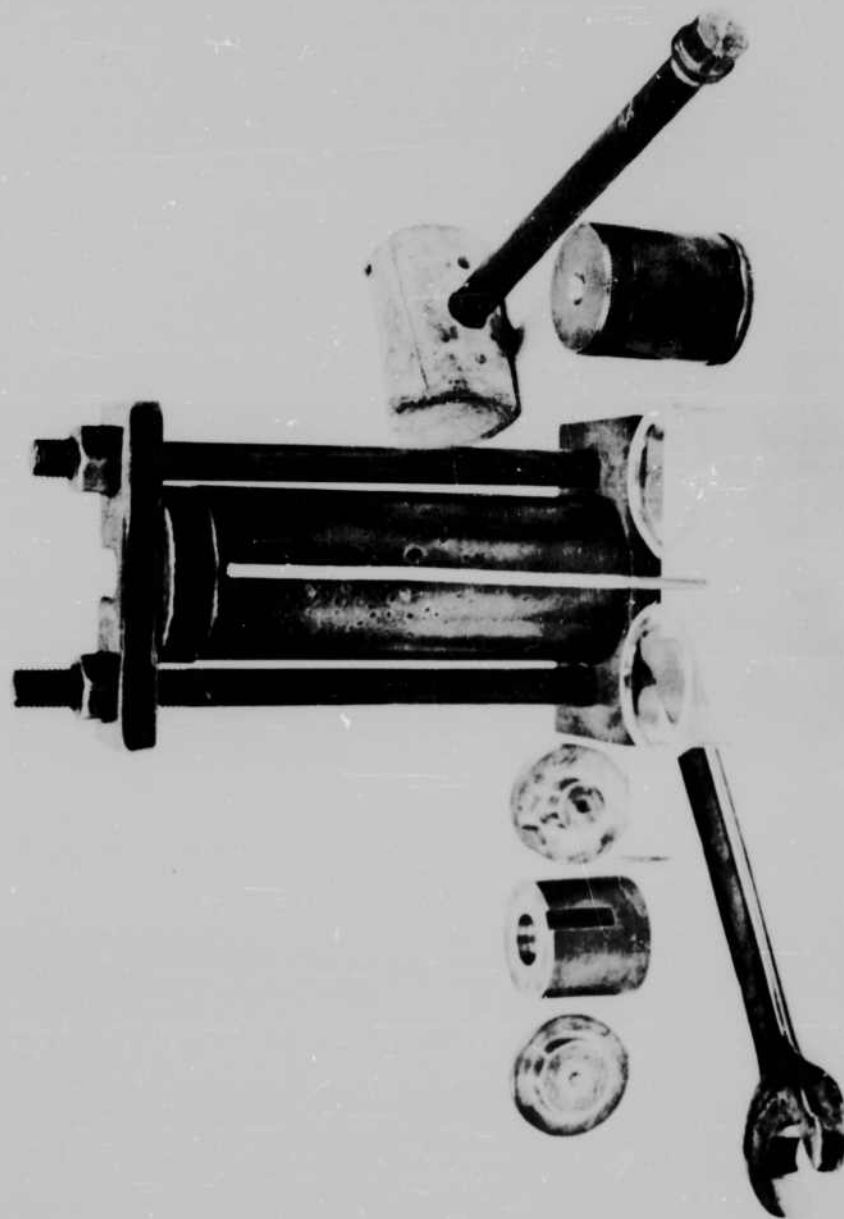
Fig 9   Components of apparatus and materials used for sand test

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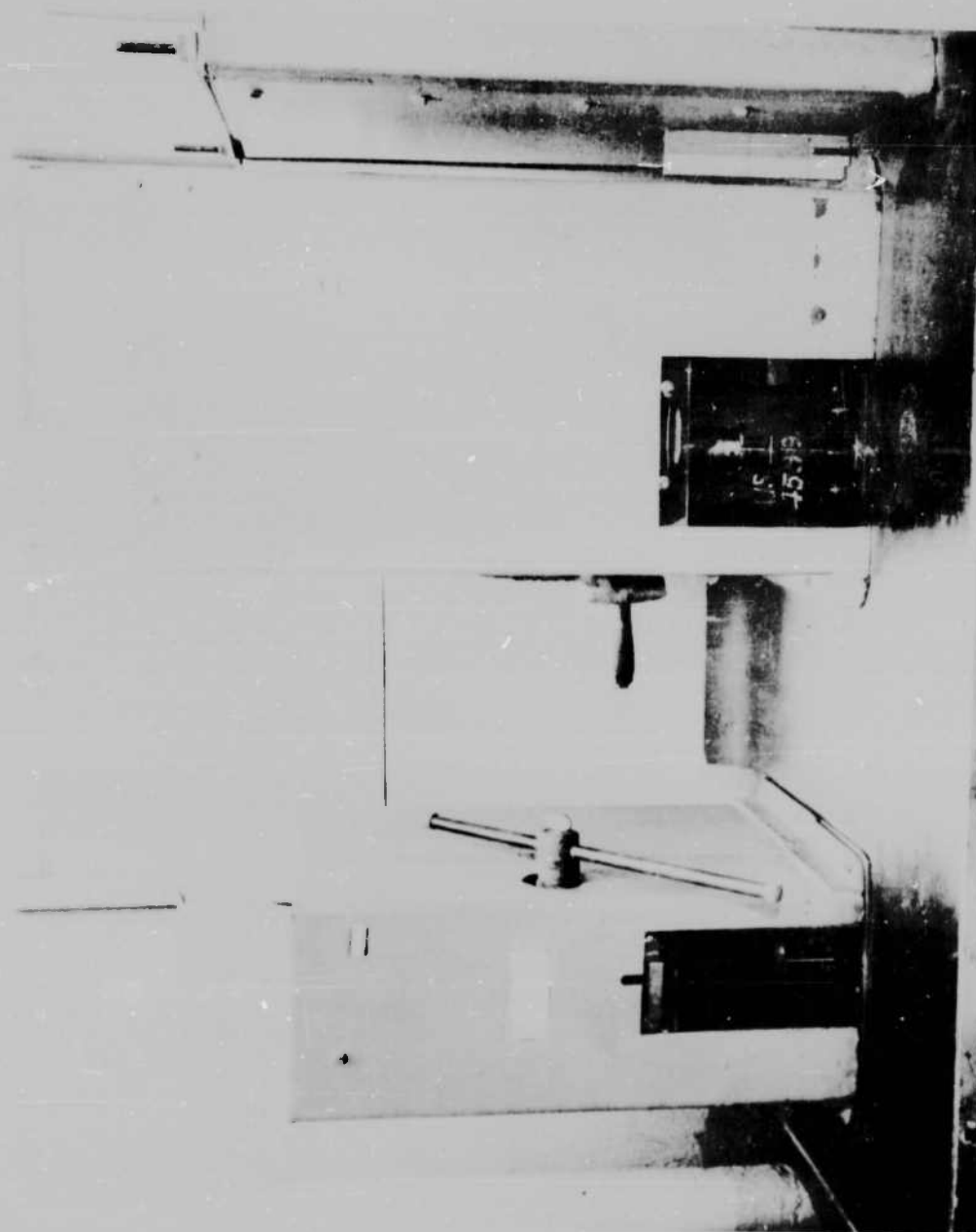
Fig 10   Presses used in sand tests

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With a pin, prick the powder train in one end of a piece of miner's black powder fuse 8 or 9 inches long. Insert the pricked end of the fuse into the crimping fixture, taking care that the end of the fuse is held firmly against the charge in the cap. Close the barricade door, and apply pressure gently to crimp the fuse in place. Load a total of 5 blasting caps in a similar manner.

Pour 80.0 ± 0.1 grams of standard Ottawa sand, which passes through a No. 20 U.S. standard sieve (opening 0.0331 inch, wire diameter 0.0165 inch) and is retained on a No. 30 U.S. standard sieve (opening 0.0232 inch, wire diameter 0.0129 inch), into the cavity of the sand test bomb (shown in Figs 9 and 11, pp 16 and 19) and level it by striking the bomb vigorously with a hammer at least 5 times while rotating the bomb on its axis. Lower the fuse and cap into the bomb cavity so that the cap is centered at the axis of the bomb and just touches the sand. Hold the cap and fuse in this position and carefully pour 120.0 ± 0.1 grams more of the sand into the bomb, being careful not to disturb the position of the cap.

To avoid possible error caused by the explosion blowing crushed sand through the hole in the cover, slip a piece of rubber tubing, about ¹⁄₄ inch long and of such inner diameter that it fits the fuse snugly, over the fuse and adjust at a point on the fuse so that the rubber will be against the inner side of the bomb cover, with the loaded cap in position as described above. Carefully insert

the fuse through the hole in the bomb cover and lower the cover into position and fasten securely. Then strike the bomb vigorously with a hammer five times while rotating the bomb on its axis.

Light the fuse and after the explosion has taken place empty the sand onto a sheet of smooth (glazed) paper taking care to remove any sand which may adhere to the sides of the bomb, the bomb cover, or to pieces of the detonator cap or burnt fuse. Transfer all of the sand to a No. 30 U.S. standard sieve fitted with a bottom pan and cover. Shake the assembly to separate the sand which has been crushed fine enough to pass through the No. 30 U.S. standard sieve. Shaking for 3 minutes on a mechanical shaker which moves back and forth horizontally 225 times per minute with a 3-inch stroke (Fig 12, p 20) has been found to be satisfactory. Weigh the sand which passes through the sieve. Record the average of the values obtained in this manner.

Press load 5 additional caps with 0.400-gram portions of the explosive in the manner described above except that the reinforcing cap is left out. Add a weighed charge of 0.300 gram of lead azide, insert a reinforcing cap, and subject the charge to a pressure of 3000 psi for 3 minutes. Determine the weight of sand crushed by each charge as directed above, and correct this by subtracting the weight of sand found to be crushed by a charge of 0.300 gram of lead azide. Record the average of the corrected values obtained in this manner.
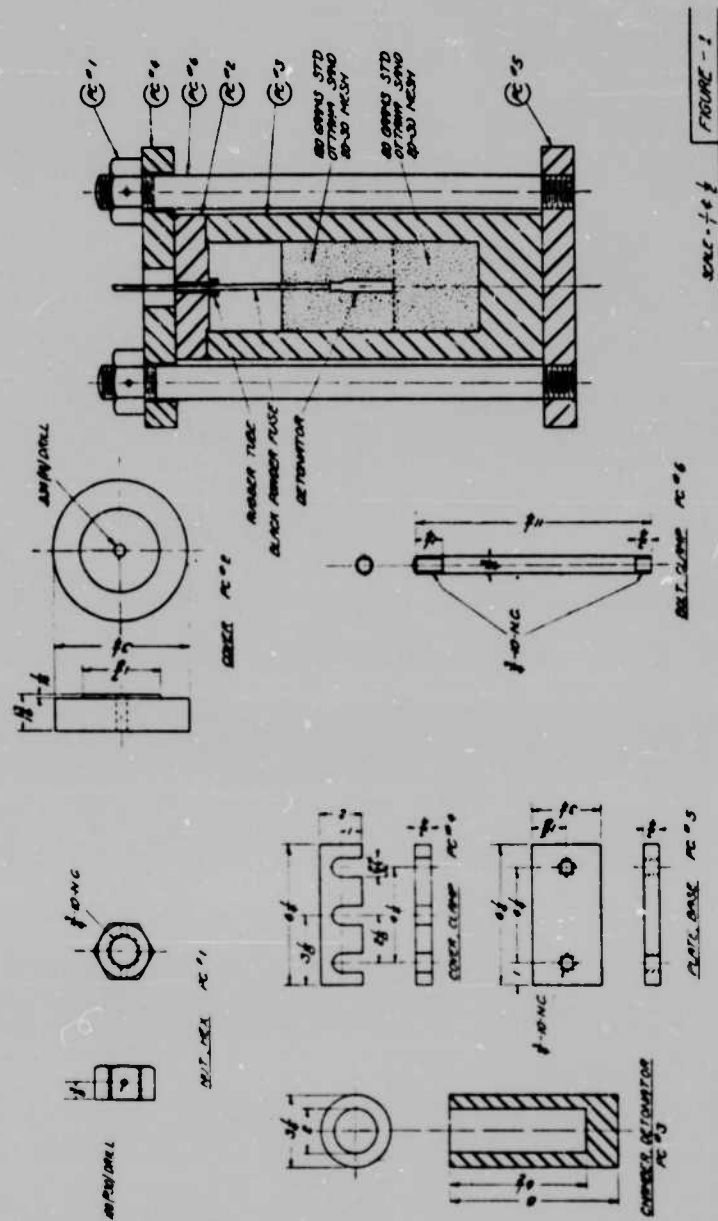
Fig 11   Details of sand test bomb

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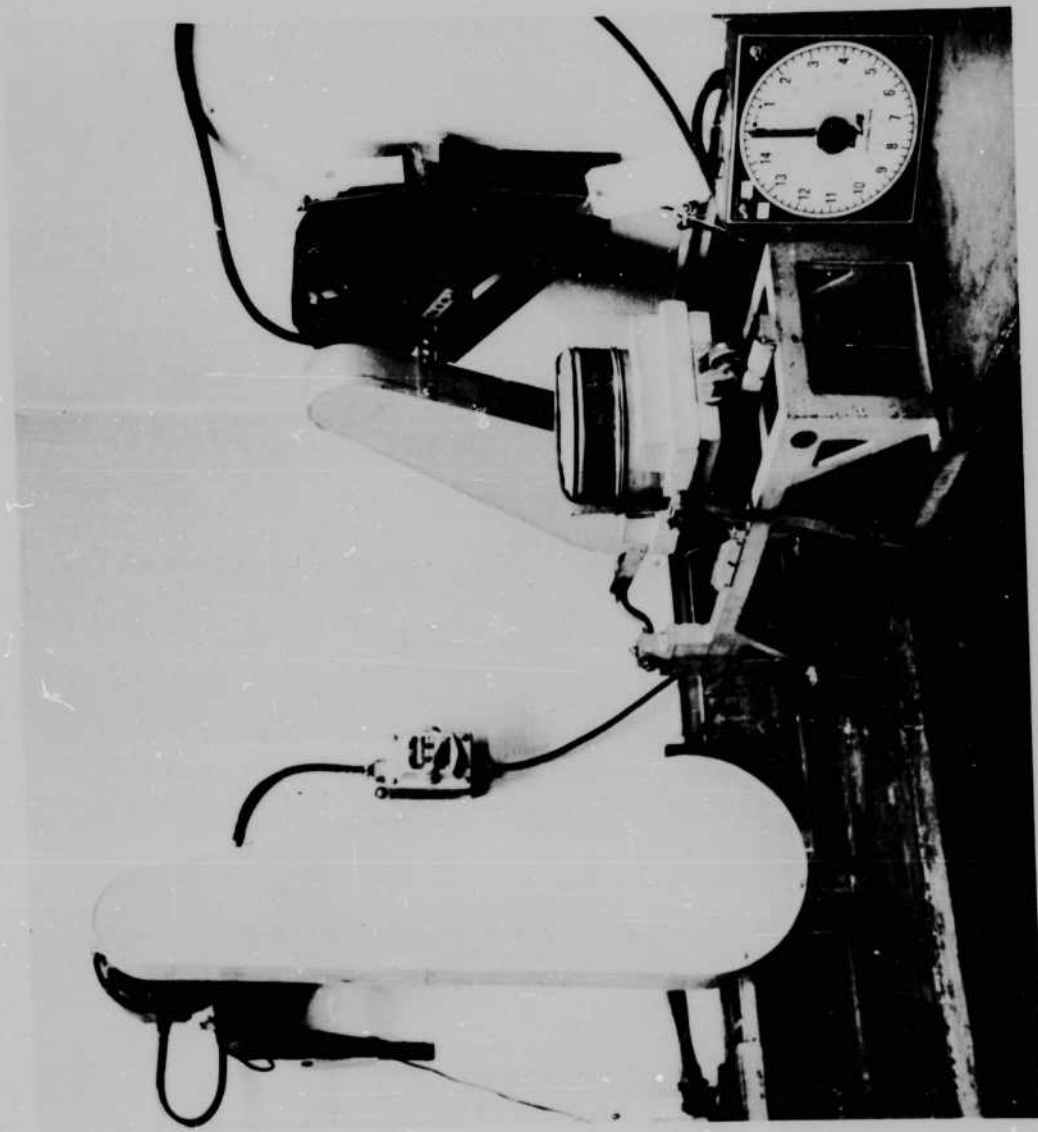
Fig 12   Sieve shaking apparatus and timer used in the sand test

If the corrected values obtained with an initiating charge of lead azide do not vary by more than 3.0 grams and the average corrected value does not exceed by more than 2.0 grams the average value obtained without an initiating charge, consider the explosive under test to be sensitive to initiation by flame alone.

If the average corrected value obtained with lead azide initiator exceeds by more than 2.0 grams the average value obtained without lead azide, press load 5 additional caps with 0.400-gram portions of the explosive in the manner described above. Add a weighed charge of 0.10 gram of tetryl and press at 3000 psi for 3 minutes. Add a weighed charge of 0.200 gram of lead azide, insert a reinforcing cap, and press at 3000 psi for 3 minutes. Determine the weight of sand crushed by each charge as directed above, and correct this by subtracting the weight of sand found to be crushed by a charge of 0.10 gram of tetryl and 0.200 gram of lead azide. Record the average of the corrected values obtained in this manner.

If the average corrected value obtained with tetryl and lead azide present does not exceed by more than 2.0 grams the average corrected value obtained with lead azide alone, repeat the test with successively smaller weights of lead azide alone. Consider as the minimum detonating charge the minimum weight of lead azide which in five tests yields corrected values which do not vary by more than 3.0 grams and which

average not more than 2.0 grams less than similar values obtained with 0.300 gram of lead azide as the initiating charge.

If the average corrected value obtained with tetryl and lead azide as the initiating charge exceeds by more than 2.0 grams the average corrected value obtained with 0.300 gram lead azide as the initiating charge, determine the minimum detonating charge of tetryl required to cause detonation of the explosive at its maximum rate. Repeat the test with successively larger or smaller weights of tetryl and 0.200-gram charges of lead azide. Consider as the minimum detonating charge the minimum weight of tetryl which in five tests yields corrected values which do not vary by more than 3.0 grams, and the average corrected value for which is not more than 2.0 grams less than the maximum average corrected value obtained with tetryl and lead azide as the initiating charge.

G. Sensitivity to Initiation by the Modified Sand Test for Liquid Explosives

Transfer weighed 0.400-gram portions of the liquid sample to be tested to each of five No. 6 commercial blasting caps composed of copper, gilding metal, or aluminum and having an internal diameter of 0.222 – 0.002 inch. Pick up each of the loaded caps with a pair of tongs and carefully transfer to the crimping fixture. With a pin, prick the powder train in one end of a piece of miner's black powder fuse 8 or 9 inches long. Crimp to the pricked end one of the caps loaded as directed above, taking care

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that the end of the fuse just contacts
the liquid sample. Crimp near the mouth
of the cap so as to avoid squeezing the
charge.

Pour 80.0 ± 0.1 grams of standard
Ottawa sand, which passes through a
No. 20 U.S. standard sieve (opening
0.0331 inch, wire diameter 0.0165 inch)
and is retained on a No. 30 U.S. standard
sieve (opening 0.0232 inch, wire diam-
eter 0.0129 inch) into the cavity of the
sand test bomb (Fig 11, p 19) and level
it by striking the bomb vigorously with
a hammer at least 5 times while rotating
the bomb on its axis. Lower the fuse and
cap into the bomb cavity so that the cap
is centered at the axis of the bomb and
just touches the sand. Hold the cap and
fuse in this position and carefully pour
120.0 ± 0.1 grams more of the sand into
the bomb, being careful not to disturb
the position of the cap.

To avoid possible error caused by the
explosion blowing crushed sand through
the hole in the cover, slip a piece of
rubber tubing, about ¼ inch long and of
such inner diameter that it fits the fuse
snugly, over the fuse and adjust at a
point on the fuse so that the rubber will
be against the inner side of the bomb
cover, with the loaded cap in position as
described above.

Carefully insert the fuse through the
hole in the bomb cover and lower the
cover into position and fasten securely.
Then strike the bomb vigorously with a
hammer five times while rotating the
bomb on its axis. Light the fuse and

after the explosion has taken place,
empty the sand onto a sheet of smooth
(glazed) paper taking care to remove any
sand which may adhere to the sides of
the bomb, the bomb cover, or to pieces of
the detonator cap or burnt fuse.

Transfer all of the sand to a No. 30
U.S. standard sieve fitted with a bottom
pan and cover. Weigh the sand which
passes through the sieve after shaking
mechanically for three minutes. Record
the average of the values obtained in
this manner.

Press load five additional No. 6 com-
mercial blasting caps with 0.300 gram
of lead azide, using a reinforcing cap,
and press the charge at 3000 psi for 3
minutes using the loading procedure
described in Section 1F (p 15).

With a pin, prick the powder train in
one end of a piece of miner's black
powder fuse 8 or 9 inches long. Insert
the pricked end of the fuse into the
crimping fixture, taking care that the
end of the fuse is held firmly against
the charge in the cap. Close the barri-
cade door and apply pressure gently to
crimp the fuse in place.

Transfer 0.400-gram portions of the
liquid explosive to each of 5 aluminum
caps. The aluminum caps should have
the following dimensions: length,
2.00 ± 0.05 inch; internal diameter,
0.248 ± 0.005 inch; wall thickness,
0.025 ± 0.001 inch. Insert the No. 6
blasting cap containing the fuse and

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lead azide into the aluminum cap until the base of the cap just contacts the liquid sample and secure by crimping the aluminum cap to the No. 6 cap.

Determine the weight of sand crushed by each charge as directed above, and correct this by subtracting the weight of sand found to be crushed by a charge of 0.300 gram of lead azide loaded in a similar assembly without the liquid explosive. Record the average of the corrected values obtained in this manner.

If the corrected values obtained with an initiating charge of lead azide do not vary by more than 3.0 grams and the average corrected value does not exceed by more than 2.0 grams the average value obtained without an initiating charge, consider the explosive under test to be sensitive to initiation by flame alone.

If the average corrected value obtained with lead azide initiator exceeds by more than 2.0 grams the average value obtained without lead azide, press load 5 additional No. 6 caps with 0.10 gram of tetryl, then add 0.200 gram lead azide along with a reinforcing cap and again subject the charge to a pressure of 3000 psi for 3 minutes, using the procedure described earlier in this report.

Attach a piece of miner's fuse to each cap. Transfer 0.300-gram portions of the liquid explosive to each of 5 aluminum caps and insert the caps containing the fuse, lead azide, and tetryl into the aluminum caps in the manner already described.

Determine the weight of sand crushed by each charge as directed above, and correct this by subtracting the weight of sand found to be crushed by a charge of 0.10 gram tetryl and 0.200 gram lead azide loaded in a similar assembly without the liquid explosive. Record the average of the corrected values obtained in this manner.

If the average corrected value obtained with tetryl and lead azide present does not exceed by more than 2.0 grams the average corrected value obtained with lead azide alone, repeat the test with successively smaller weights of lead azide alone. Consider as the minimum detonating charge the minimum weight of lead azide which in 5 tests yields corrected values which do not vary by more than 3.0 grams and the average corrected value for which is not more than 2.0 grams less than the average corrected value obtained with 0.300 gram of lead azide as the initiating charge.

If the average corrected value obtained with tetryl and lead azide as the initiating charge exceeds by more than 2.0 grams the average corrected value obtained with 0.300 gram lead azide as the initiating charge, determine the minimum detonating charge of tetryl required to cause detonation of the explosive at its maximum rate. Repeat the test with successively larger or smaller weights of tetryl and 0.200-gram charges of lead azide. Consider as the minimum detonating charge the minimum weight of tetryl tests in which 5 yields corrected values which do not vary by more than 3.0 grams, and the average corrected

value for which is not more than 2.0 grams less than the maximum average corrected value obtained with tetryl and lead azide as the initiating charge.

2. BRISANCE TESTS

A. Sand Test

The brisance (shattering power) of an explosive is determined by means of the sand test.

This is done in the manner described in Section 1F (p 15), by initiating 0.400-gram charges of the explosive under test by means of

1. Flame from a fuse
2. 0.300 gram of lead azide
3. 0.100 gram of tetryl and 0.200 gram of lead azide
4. 0.250 gram of tetryl and 0.200 gram of lead azide

Consider as the brisance the maximum average corrected value obtained. If the explosive is sensitive to initiation by flame, record also the average of the values obtained with such initiation.

B. Modified Sand Test to Determine the Brisance of Liquid Explosives

Press load, in the manner described in Section 1F (p 15), 5 No. 6 commercial blasting caps made of copper, gilding metal, or aluminum and having an internal diameter of 0.222 – 0.002 inch, with first 0.25 gram of tetryl and on top of this 0.20 gram of lead azide, using

a pressure of 3000 psi, and a 3-minute dwell to consolidate each charge.

With a pin, prick the powder train in one end of a piece of miner's black powder fuse 8 or 9 inches long. Insert the pricked end of the fuse into the crimping fixture, taking care that the end of the fuse is held firmly against the charge in the cap. Close the barricade door and apply pressure gently to crimp the fuse in place.

Transfer a weighed portion of 0.400 gram of the liquid sample to be tested to each of 5 aluminum caps. The aluminum caps should have the following dimensions: length, 2.00 · 0.05 inch; internal diameter, 0.248 · 0.005 inch; wall thickness, 0.025 · 0.001 inch. Insert the cap containing the fuse and initiator into the aluminum cap until the base of the cap just contacts the liquid sample, and secure by crimping the aluminum cap to the No. 6 cap. Then fire the assemblies in the sand test bomb in the manner described in the section of this report on "Sensitivity to Initiation as Determined by the Sand Test" and take as the brisance the average corrected value obtained (Ref 2).

C. Determination of Initiating Efficiency by Sand Test

Using 0.400-gram charges of tetryl and 0.300-gram charges of the initiating explosive, conduct the sand test as directed above. Correct each value by substracting the weight of sand crushed by 0.300 gram of the initiating explosive

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alone. If the corrected values for tetryl do not vary by more than 3.0 grams, repeat the test using successively smaller weights of the initiating explosive.

Consider as the minimum detonating charge of the initiating explosive the minimum weight of this explosive which in 5 tests yields corrected values for tetryl which do not vary by more than 3.0 grams and the average of which is within 2.0 grams of the average corrected value for tetryl obtained with 0.300 gram of the initiating explosive under test.

The following data is reported:

1. Brisance of 0.300 gram of initiator when subjected to flame
2. Brisance of 0.400 gram of tetryl when fired with 0.300 gram of initiator under test
3. Minimum detonating charge of initiator

To compare the initiating efficiency of the explosive under test with that of lead azide, repeat the entire procedure described above using lead azide as the initiator. Compare the values obtained with lead azide and the initiator under evaluation to determine the initiating efficiency of the latter.

3. STABILITY TESTS

Stability tests in general measure the resistance of explosives to decomposition by heat, the end point sometimes being the change in color of a special test paper or the evolution of oxides of nitrogen (red fumes). In other cases, the loss in weight or the volume of gas liberated is measured after a standard heating period. In one test, the degree of hydrolysis at an elevated temperature is determined after a definite period.

Stability tests applicable to one type of explosive generally are not suitable for use with other types. The types described and their uses are as follows:

Test	Type of Explosive
75°C International test	High explosives and pyrotechnics
82.2°C KI test	High explosives and pyrotechnics
100°C Heat test	High explosives and pyrotechnics
90°C Vacuum stability test	High explosives and pyrotechnics; also propellants
100°C and 120°C Vacuum stability tests	High explosives and pyrotechnics
65.5°C Surveillance test	Propellants
80°C Surveillance test	Propellants
120°C Heat test	Propellants
134.5°C Heat test	Propellants; also nitrocellulose
Taliani test	Propellants; also nitrocellulose
65.5°C KI test	Nitrocellulose
240-Hour hydrolysis test	Nitrocellulose

75°C International Test

Transfer a 10-gram portion of the sample as received to a weighing bottle 35 mm in diameter and 50 mm high, cover this with a watch-glass, and weigh. Heat the covered bottle and contents at 75°C for 48 hours, cool in a desiccator, and weigh. Calculate the

loss in weight to percent and report as volatility of the sample.

Note whether the material has undergone decomposition or is markedly volatile, as indicated by:

1. Discoloration

2. Appearance of colored fumes above the material

3. Development of an odor indicating acidity

4. A loss of weight distinctly in excess of moisture content (as determined by a method appropriate to the material)

82.2°C KI Test

This test, used generally for nitroglycerin, is conducted in the same manner as the 65.5°C KI test (see below), except that the temperature of the bath is maintained at 82.2° ± 0.1°C and a 2-cc sample is used.

100°C Heat Test

Determine the moisture content of the sample by drying a weighed 10-gram portion in a desiccator until constant weight is reached. Calculate the percentage of moisture in the sample.

Transfer a weighed portion of 0.6 gram of the sample as received to each of 2 tared test tubes 75 mm in length and 10 mm in diameter and to each of 2 similar but untared test tubes. As a safety measure, place each set of tubes in a pipe bomb (which is a capped pipe

2 inches in diameter and about 4 inches long and vented by means of four ¼-inch holes drilled around the center, 90° apart). Screw the top cap in place and place the assembly in an oven maintained at 100° ± 1°C.

After 48 hours, remove the pipe bomb containing the 2 tared tubes. Allow it to cool for a few minutes, then remove the tubes, cool them in a desiccator, and weigh them. Replace the tubes in the bomb, replace the bomb in the oven, heat it for another 48 hours, remove it as previously described, cool it in a desiccator and reweigh it. Calculate the percentage loss in weight during each 48-hour period of heating and subtract the percentage of moisture from the weight lost during the first heating period.

Allow the 2 untared tubes to remain in the oven for 100 hours of continuous heating and note whether ignition or explosion occurs.

90°C, 100°C and 120°C Vacuum Stability Tests

Apparatus

The glass heating tube and glass capillary tube are shown in Figure 13 (p 27). The heating block is illustrated in Figure 14 (p 28) and is described in detail in Figure 13.

Procedure

Adjust the temperature of the block heater so that the variation in the sample

26. C M.

2.8

8 CM 1.5 CM

15.5 CM

14. CM

1.5 CM

1.5 CM

STOPPER TO BE
VERY WELL GROUND.

85. CM.

CAPILLARY TUBING
6. TO 6.5 MM. EXTERNAL
DIA., 1.5 TO 2. MM.
INTERNAL DIA.

45°

2.2 CM

8. CM

Fig 13   Glass heating tube and glass capillary tube for 120°C vacuum stability test

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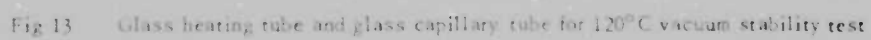
Fig 14   Heating block for vacuum stability test

within the heating tube is within ±0.5°C of the desired test temperature. Determine the temperature within the heating tubes by inserting a calibrated thermometer to the bottom of one of the tubes specified for the test and then inserting the assembly into one of the block wells. Apply the necessary exposed mercury column and bore corrections to the observed temperature reading.

Transfer 5.00 grams of the dried sample (or 1.00 gram of initiating explosives, or explosives of similar sensitivity) to the heating tube of the apparatus shown in Figures 13 and 14. Connect the capillary tube to the heating tube. Clamp the apparatus so that the long section of the capillary tube is in a nearly vertical position and the lower end rests on a solid support.

Transfer 7.0 cc of mercury to the cup at the lower end of the capillary tube. By means of a vacuum pump, evacuate the system until the pressure is reduced to approximately 5 mm of mercury. If the apparatus is tilted forward until the capillary opening of the cup is free of mercury, evacuation will be facilitated.

Disconnect the pump, seal the connections by means of 1 cc of mercury, and measure the total vertical height of the column of mercury in the capillary tube. Measure and subtract the vertical height of the column of mercury in the cup. This difference is the value (H_1). Note the room temperature (t_1) and barometric pressure.

Insert the heating tube, assembled to the capillary tube as described above, into one of the wells of the block heater and heat for 40 hours. Remove the heating tube and allow it to cool to room temperature. Measure the total vertical height of the column of mercury in the capillary tube and subtract from it the vertical height of the column of mercury in the cup. This difference is the value (H). Note the room temperature (t) and the barometric pressure. Calculate the volume of gas, at standard temperature and pressure, liberated during the test.
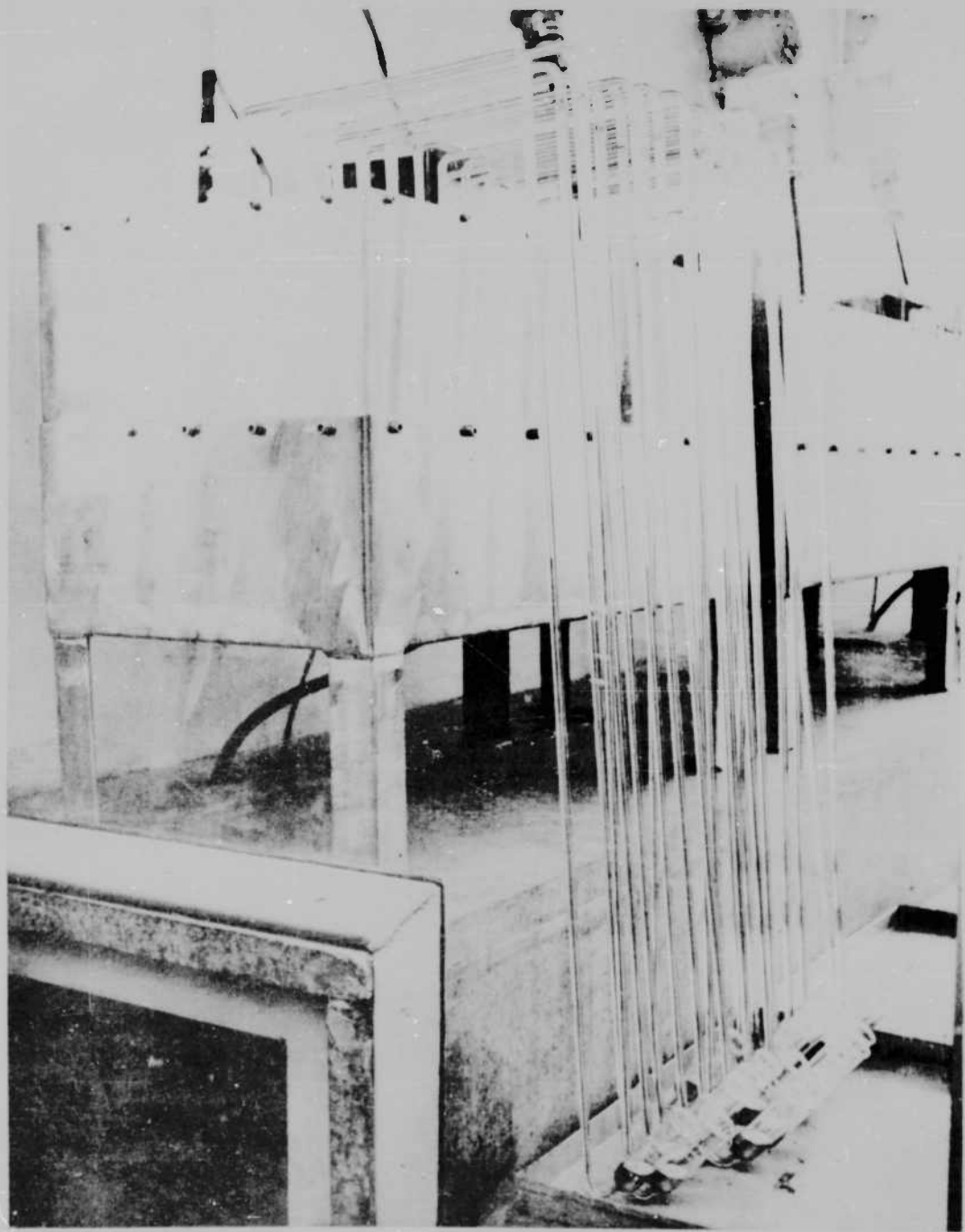
$$cc \ gas = A + C (B - H) \ \frac{273P}{760 (273 + t)} -$$

$$A + C(B_1 - H_1) \frac{273 P_1}{760 (273 + t_1)}$$

where:

A is the volume of the heating tube minus 5.00 cc or 1.00 cc (volume of explosive)

B_1 is the total length in mm of the capillary tube minus the vertical height in the column of mercury in the cup before heating

B is the total length in mm of the capillary tube minus the vertical height of the column of mercury in the cup at the end of the test

C is the determined unit capacity of the capillary tubing 'i cc per mm

P_1 is barometer reading when the test is started minus H_1

P is barometer reading at the end of the test minus H.

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Calibration of the Glass Tube

Determine the volume in cubic centimeters of the 15.5-cm heating tube by running in mercury from a burette until the tube is filled to the level at which the ground glass joint of the capillary tube makes contact with the mercury. Transfer 7.0 cc of mercury to the cup at the lower end of the capillary tube, clamp the tube in a vertical position, and measure the height, in millimeters, of the mercury column in the capillary tube. Measure the length, in millimeters, of each of the 3 parts of the capillary tube, add these values, and subtract the height of the mercury column (approximately 25 mm) determined as directed above. Determine the capacity of the capillary tube per unit of length as follows:

Transfer an accurately weighed portion of approximately 10 grams of mercury to the cup at the lower end of the capillary tube. Manipulate the tube so that, when it is horizontal, mercury is contained in a continuous section of the longest part of the tube, and then measure the length of the mercury column. Repeat this twice with the mercury in 2 other parts of the longest section of the tube. Calculate the average of the 3 measured lengths of the mercury column and calculate the volume of the capillary tube.

$$cc \text{ per mm} = \frac{W}{13.59L}$$

Where:

W = the weight in grams of the mercury

L = the average length in millimeters of the mercury column

65.5°C Surveillance Test

For this test, use a thoroughly washed and dried 8-ounce glass-stoppered bottle having a wide mouth with a velvet-ground mushroom stopper which provides an essentially air-tight seal. The bottle must be etched or ground on the side (1 × 2 inches) so as to be suitable for labeling and must be made from a special resistance glass which has practically no color and an alkalinity of not more than 0.02% (calculated as potassium hydroxide). Determine the alkalinity of the ground glass by placing a 10-gram sample in a Pyrex flask, adding 20 cc of N/20 hydrochloric acid, allowing to stand for 24 hours, and titrating with N/20 sodium hydroxide, using phenolphthalein indicator.

Transfer a 45-gram portion of the sample to the bottle. If the propellant is for 10-inch or larger cannon, use a test sample of 5 propellant grains or as many as can be inserted in the bottle. For rocket propellants, prepare the grains, or sheets, in the following manner:

For small diameter sticks, cut a sufficient number of 3-inch lengths.

For large diameter sticks (up to and including 1¾ inches), cut a sufficient amount from the length.

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For larger cylindrical sticks (more than 1¾ inches in diameter) cut a longitudinal section through the center, 1 inch wide on the grain surface, 1¼ inches in depth, and of sufficient length.

For larger cruciform sticks, cut a longitudinal section from one arm, ¼ inch wide on the exterior grain surface and ¼ inch deep on the recession, and of sufficient length. Include lateral inhibitor, if present.

For sheet powders, cut sufficient lengths 2½ inches wide, and coil.

Stopper the bottle by simultaneously twisting and pressing on the stopper. Label the bottle so as to show the lot number of the propellant and the date on which the test is begun.

Place the bottle and contents in a chamber maintained at $65.5° \pm 1.0°C$ (see Fig 15, p 32), the air in which is preferably maintained in constant circulation. After the bottle and contents have been heated for 24 hours, reseat the stopper. Make daily observation of the appearance of the bottle and contents. Note the number of days required to cause the liberation of visible reddish fumes of oxides of nitrogen.

It has been customary to consider any propellant having a test value of 90 days or less to be of definitely impaired stability and any propellant having a test value of 20 days or less to be sufficiently unstable to require destruction or salvage.

80°C Surveillance Test

This test is conducted in the same manner as the $65.5°C$ surveillance test, except that the temperature of the chamber is maintained at $80° \pm 1.0°C$. This test is used in conjunction with the $65.5°C$ surveillance test to obtain anticipatory data.

120°C and 134.5°C Heat Tests

Procedure

Make 5 tests of 5 portions of the sample in either the 120°C or 134.5°C heat test in the following manner:

Prepare a constant temperature bath (PA Dwg. CP 24114), containing a solution of glycerin and water and having a specific gravity of approximately 1.21, which will maintain a temperature of $120° \pm 0.5°C$ in the heating tubes, or a similar solution, having a specific gravity of approximately 1.24, which will maintain a temperature of $134.5°C \pm 0.5°C$ in the heating tubes. An aluminum block bath which will maintain temperatures within these tolerances is recommended to anyone setting up this test. Determine the temperature within the heating tubes by inserting a calibrated thermometer to the bottom of one of the tubes specified for the test and inserting the tube to the bottom of one of the bath wells. Apply the necessary exposed mercury column and bore corrections to the observed temperature reading.

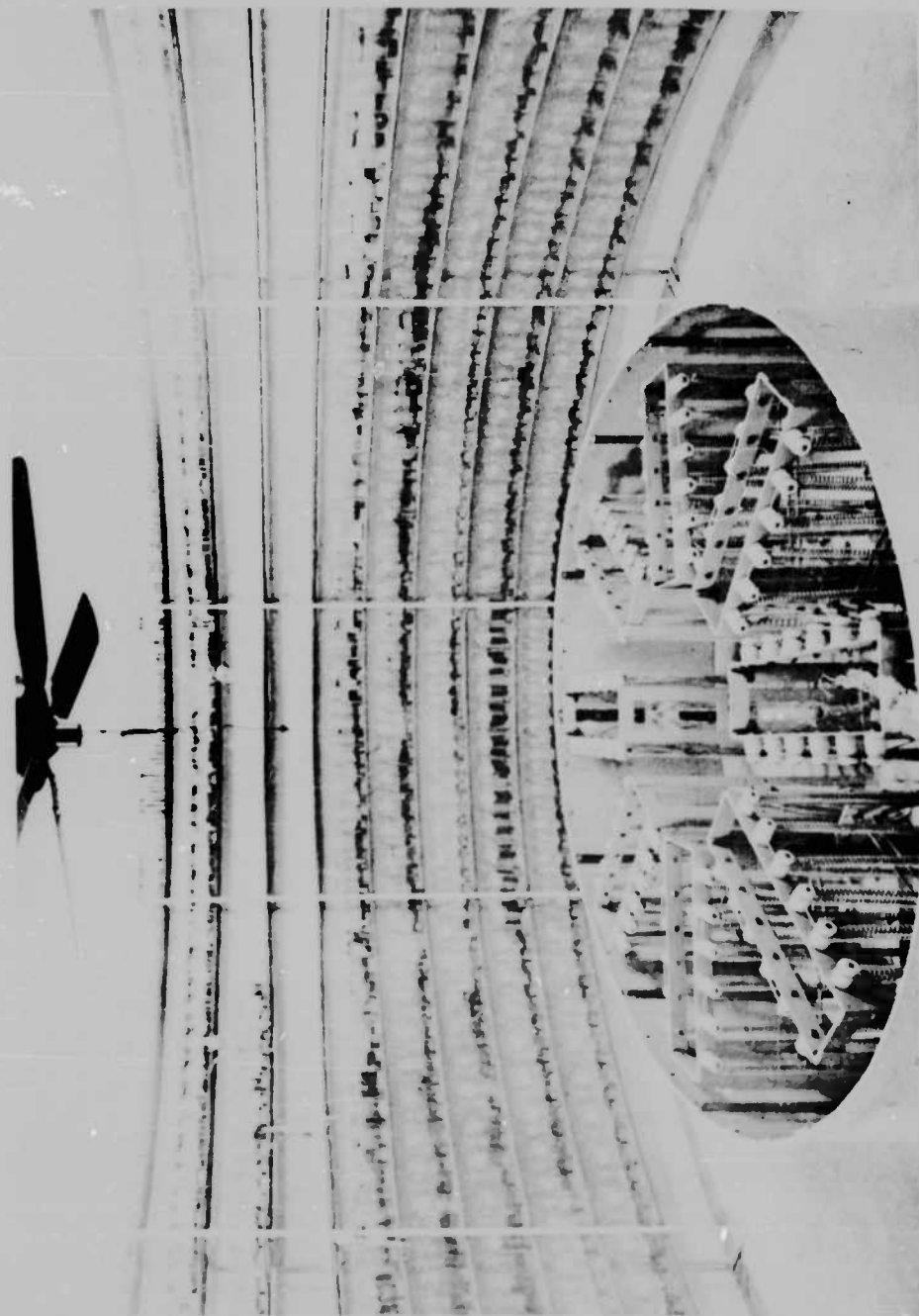Weigh out several 2.5-gram portions of the sample and place each in a test

Fig 15   65.5°C surveillance test chamber

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tube made of heavy glass, preferably Pyrex, approximately 15 mm inside diameter, 18 mm outside diameter, and 290 mm long. Each portion should consist as nearly as possible of whole grains, consistent with the 2.5-gram weight requirement for the sample. In sectioning grains to obtain the required weight, split the grains longitudinally. Place a piece of standard, normal methyl violet paper,[1] 70 mm long and 20 mm wide, vertically in each tube with its lower edge 25 mm above the test material. Stopper each tube with a cork through which a hole 4 mm in diameter has been bored or which has been notched to a depth of approximately 4 mm. Place 5 of the tubes in the approximately constant temperature bath, no more than 6 or 7 mm of tube length should project from the bath. Examine each tube at 5-minute intervals by withdrawing one-half of its length and replacing it quickly. Record as the salmon pink test value the time of observation which reveals the test paper in any tube to be completely changed to a salmon pink color.

After the salmon pink values have been determined, heating of the tubes is continued and the time of observation which reveals any of the tubes to be completely filled with red fumes is recorded.

After the red fume value has been determined, heating is continued further

[1]Standardized in accordance with Method 404.2 (T) of MIL-Std-286A.

to determine whether any sample of propellant will explode in less than 5 hours.

When the 134.5°C heat test is used in testing nitrocellulose, only two tubes are processed. Before testing, place the sample on a paper tray and dry it for 4 to 5 hours at $40 \pm 2°C$, for 1 to $1\frac{1}{2}$ hours at $50 \pm 2°C$, or overnight at room temperature with further drying for $\frac{1}{2}$ hour at $40° \pm 2°C$. Weigh out several 2.5-gram samples and press them into the lower 2 inches of the tubes. Process as directed above, with the first observation at 20 minutes, and subsequent observations at 5-minute intervals thereafter. Discontinue the test when the salmon pink end point is reached.

Taliani Test

Apparatus (See Fig 16)

Heating Block. The heating block is a machined aluminum cylinder wound with three coils of resistance wire. The coils are insulated from the block with micabond and from each other with asbestos cord. The temperature is maintained and controlled by a Fenwal thermoregulator operating through an electronic circuit. A Fisher No. 15-000 thermometer (99° to 200°C, $\frac{1}{10}$°C div, 60 mm) is used.

Insulation of the block is accomplished by supporting it on transit strips inside a cylindrical steel container, and packing the space between the block and the cylinder with glass wool. The top of the
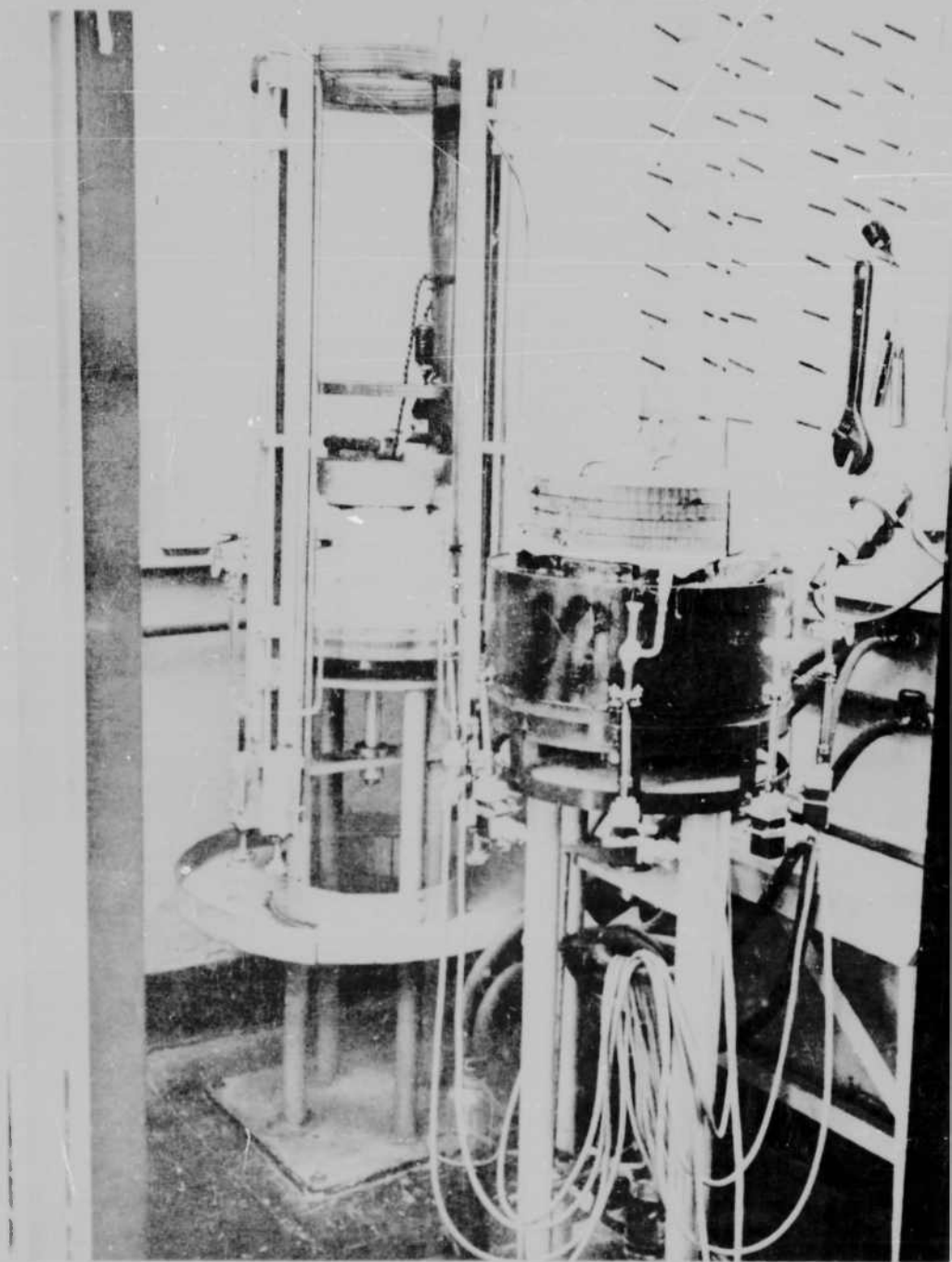
Fig. 16   Taliani test apparatus

block and the head of the Fenwal thermo-regulator are further insulated by painting the surface of the block with a heat-insulating material and fastening a $\frac{1}{4}$-inch Synthane ring to the periphery of the block. This ring supports two $2\frac{5}{8}$-inch-thick semicircles of fire brick which have been cut to clear the Fenwal head and thermometer. The fire brick is painted with a heat-resistant paint. A pad of glass wool is placed between the block and the fire brick for additional insulation.

Glassware. Supports for the manometer and connecting glassware are constructed so as to have 2 degrees of freedom. This arrangement permits more flexible use of the glassware with good assurance of obtaining gas-tight joints. The glassware used should be standardized by bringing the total volume of the closed system to 6.00 ml. This is accomplished as follows: The volume of the helix is cal-ibrated directly by introduction of mercury from a burette, and each sample tube is numbered and calibrated directly for volume with mercury from a burette. The volume is usually slightly more than 6.00 ml and is adjusted by using an appropriate number of 3 mm glass beads. Each lot of beads is statistically "sized" by measuring the mercury displaced by 100 beads. The beads are introduced into a partially filled burette in increments of 10 (for establishment of uniformity within the lot) and the volume per bead is cal-culated. The glass-to-metal joint between the manometer and the mercury reservoir is made by employing an adapter, $\frac{1}{2}$ inch in length, cut from $\frac{1}{4}$-inch, thin-walled,

soft rubber tubing. The end of the mano-meter is moistened, inserted half way into the adapter, and the junction is effected by pressing the adapted mano-meter into the mercury reservoir recess with a downward and circular motion.

Nitrogen Infusion-Evacuation Apparatus. An adapter fitted to the connector tube exhaust is connected to a 2-way stop-cock, which in turn is connected to (1) a nitrogen tank equipped with pressure gage, needle valve, and bubble counter containing dibutylphthalate; and (2) a vacuum system equipped with modulating control.

Procedure

Rasp or grind the propellant in a Wiley Mill to 10 to 40 mesh and transfer 1.000 ± 0.001 gm to a sample tube. Run all samples in duplicate. Fit the tube to the helix and place it in the block, which is normally at $110° \pm 0.1°C$. At-tach the helix and the manometer to the connector tube, using silicone stopcock grease in all glass-to-glass joints. Con-nect the connector tube exhaust to the nitrogen infusion-evacuation apparatus, which employs a 2-way stopcock and is closed to the atmosphere.

By adjustment of the 3-way stopcock of the connector tube exhaust, close off the manometer and alternately evacuate and infuse with nitrogen 5 times. (In-fusion of nitrogen is controlled by the bubble counter, in which the rate of flow of the gas produces bubbles too rapidly to count but not so rapidly as to cause

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undue agitation). Adjust the connector tube stopcock to closed position. Disconnect the nitrogen infusion-evacuation apparatus from the connector tube exhaust.

After a 15-minute induction period, open the upper system to the atmosphere in order to attain atmospheric pressure. Immediately adjust the connector tube stopcock to close the upper system and open the manometer to the atmosphere. Raise the mercury level connector tube stopcock; adjust the stopcock to close the total system to the atmosphere and open the manometer to the upper system. Adjust the mercury to the fiducial mark and observe the mercury level in the manometer side arm. Record the block temperature and barometric pressure with each manometer pressure and time reading (every 30 minutes after the induction period).

Add each observed reading to the barometer reading for that particular time, and subtract the zero point for that unit from the sum. The difference represents the absolute pressure of the system. The first reading (at atmospheric pressure) after the 15-minute induction period serves as the reference point. Subtract this reference point from each subsequent absolute pressure, and record the difference as Δp, with a subscript identifying the unit. The Δp values for the duplicate tubes on any individual propellant lot should agree within 4 mm in the 0-50 mm range, 6 mm in the 50-100 mm range, and 8 mm above 100 mm.

Average the two values of Δp and plot the average Δp on the ordinate, and the time in minutes on the abscissa of 10×10 graph paper. Report slope at 100 mm, minutes to 100 mm, and slope at 100 minutes.

65.5°C KI Test

Conduct this test in an atmosphere free of oxides of nitrogen, acids or any other vapors which might significantly affect the test results.

If the sample of nitrocellulose contains a large excess of water, press it in a clean cloth or wring it out in a wringer. Rub out the cake in the cloth until fine, taking care that it does not come into contact with the hands, spread out on clean paper trays, and dry in an air-bath at 35° to 40°C until the desired moisture content has been obtained. The desired moisture content is that which will give condensation of moisture in diminishing amounts in the 5 test tubes used, ranging from an appreciable amount in the first tube to none in the fifth as the test progresses. The proper condition of the nitrocellulose for the first sample is indicated when the nitrocellulose clings, after rubbing, to a spatula or to tissue paper which has been wrapped about the fingers.

Weigh out a 1.3-gram sample on a dispensing balance and transfer it to a thoroughly clean and dry tube. Weigh out other samples from the material during the drying process and place them

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in additional tubes at intervals of approximately 5 minutes until 5 tubes have been prepared. Use tubes 5.5 inches long, of 0.5-inch internal diameter and 0.62-inch external diameter.

During the test, close each tube by means of a clean, tightly fitting cork stopper through which passes a tightly fitted, thoroughly clean and dry glass rod equipped with a platinum holder for a strip of KI starch paper.[1] Discard the corks after each test. Press down or shake the nitrocellulose in each tube until it occupies a space 1.6 inches long. Hang on the platinum holder a strip of the test paper about 1 inch long and 0.37 inch wide. Moisten the upper half of the test paper with a 50% solution of pure glycerin in water by means of a metal or glass rod, avoiding an excess and ensuring that the line of demarcation will be as even as possible. Regulate the temperature of the heating bath at 65.5°C ± 1.0°C (See Fig 17, p 38). Locate the bath in a bright reflected light, preferably that obtained through a north window. In the absence of daylight of sufficient intensity, conduct the test using artificial daylight which may be obtained with "daylight" fluorescent tubes or "daylight" incandescent bulbs.[2] Immerse each tube in the bath to a depth of 2.25 inches. Note the time when the tubes are placed in the bath.

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[1] The KI starch paper is obtained from the Naval Propellant Plant, Indian Head, Md.

[2] "Daylight" glass is manufactured by the Corning Glass Works, Corning, N.Y.

As the test proceeds, keep the line of demarcation between wet and dry test paper level with the lower edge of the moisture film on the wall of the tube. Consider the first appearance of discoloration of the damp portion of the test paper as the end of the test for each tube, and consider the minimum test obtained as the test value of the sample. Disregard any discoloration not greater than that obtained at the same time by a blank test.

Standard grades of military nitrocellulose are required to have a minimum 65.5°C KI test value of 35 minutes.

This test is also used in connection with the acceptance of Cordite N and Cordite N/M propellants, and experimentally in the study of any propellant. For such testing the Cordite N and Cordite N/M propellants are ground in a suitable hand-power utility mill such as that designated as item 8-450 in Fisher Scientific Company Catalog No. 90. The ground propellant is then screened and the portion which passes through 20 mesh and is retained on 40 mesh is used in the test. The testing procedure is otherwise the same as for nitrocellulose, except that no moisture series is used.

240-Hour Hydrolysis Test

The 240-hour hydrolysis test shall be run in duplicate and the average of the two determinations reported.

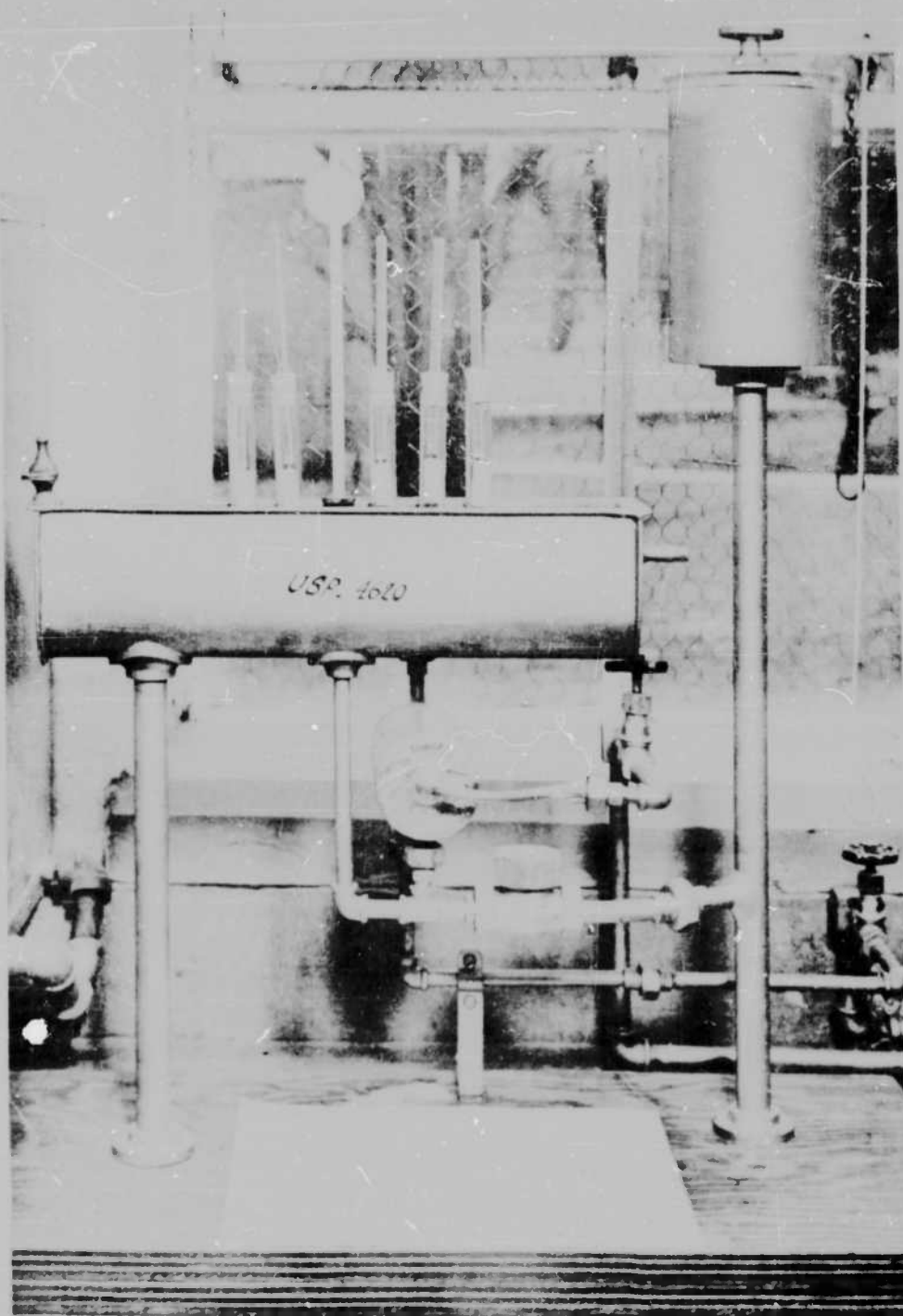Weigh a 5-gram sample of nitrocellulose which has been air-dried for 24 hours,

Fig. 17   Apparatus for 65.5° C KI test

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and the moisture content of which has been determined. Transfer the weighed sample of nitrocellulose to a 250-cc Pyrex glass flask which is connected by means of a ground glass joint to a Pyrex condenser. Add 100 cc of distilled water and connect the flask to the condenser. Place the flask in a water bath in which the water is kept constantly boiling. Remove the flask from the water bath at the end of 240 hours.

Decant the liquid from the nitrocellulose while hot, keeping as much nitrocellulose as possible in the hydrolysis flask. Filter the decanted liquid through a filter paper and collect it in a 500-cc volumetric flask. Wash the nitrocellulose twice with 75-cc portions of distilled water. Decant the wash waters and filter them through the original paper. Collect the wash waters in the 500-cc volumetric flask containing the hydrolysis solution. Wash any nitrocellulose that has accumulated on the paper back into the 250-cc hydrolysis flask.

Cover the nitrocellulose with a total of 100 cc of distilled water, stopper the flask tightly, and allow to stand overnight. Filter the third wash water into the volumetric flask, at the same time transferring the nitrocellulose to the filter paper. Wash the nitrocellulose on the filter paper until the volume of the combined washings and hydrolysis liquor reaches 500 cc. After each washing, allow the funnel to drain completely before adding more distilled water.

Shake the volumetric flask to insure uniformity of the liquid. Determine the pH of the blended solution by means of a quinhydrone electrode according to the standard electrometric procedure.

Convert the pH value of the solution into an expression of the normality of the solution by means of Table 1 (p 40).

Calculate the percentage of available nitric acid liberated by using the following equation:

$$\% \text{ of available nitric acid liberated} = \frac{\text{normality of solution} \times 3150}{\text{grams of nitric acid available in sample}}$$

Calculate the grams of nitric acid available from the nitrogen in the 5 grams of nitrocellulose by means of the following equation:

$$\text{Grams of available nitric acid} = \text{percentage of nitrogen in sample} \times 0.225.$$

REACTIVITY TEST

Concept

The contact of an explosive with a contiguous material may, in time, cause the chemical deterioration of the explosive. This may lead to the development of non-standard or hazardous conditions, or to spontaneous ignition of the explosive material. The reactivity test, by subjecting intimate mixtures of the

TABLE 1

Hydrogen-Ion Concentrations Corresponding to pH n.00–pH (n + 1).00

pH	[H·]	pH	[H·]	pH	[H·]
n.00	1.00×10^{-n}	n.34	0.46×10^{-n}	n.68	0.21×10^{-n}
n.01	0.98×10^{-n}	n.35	0.45×10^{-n}	n.69	0.20×10^{-n}
n.02	0.96×10^{-n}	n.36	0.44×10^{-n}	n.70	0.20×10^{-n}
n.03	0.93×10^{-n}	n.37	0.43×10^{-n}	n.71	0.20×10^{-n}
n.04	0.91×10^{-n}	n.38	0.42×10^{-n}	n.72	0.19×10^{-n}
n.05	0.89×10^{-n}	n.39	0.41×10^{-n}	n.73	0.18×10^{-n}
n.06	0.87×10^{-n}	n.40	0.40×10^{-n}	n.74	0.18×10^{-n}
n.07	0.85×10^{-n}	n.41	0.39×10^{-n}	n.75	0.18×10^{-n}
n.08	0.83×10^{-n}	n.42	0.38×10^{-n}	n.76	0.17×10^{-n}
n.09	0.81×10^{-n}	n.43	0.37×10^{-n}	n.77	0.17×10^{-n}
n.10	0.80×10^{-n}	n.44	0.36×10^{-n}	n.78	0.17×10^{-n}
n.11	0.78×10^{-n}	n.45	0.35×10^{-n}	n.79	0.16×10^{-n}
n.12	0.76×10^{-n}	n.46	0.35×10^{-n}	n.80	0.16×10^{-n}
n.13	0.74×10^{-n}	n.47	0.34×10^{-n}	n.81	0.15×10^{-n}
n.14	0.73×10^{-n}	n.48	0.33×10^{-n}	n.82	0.15×10^{-n}
n.15	0.71×10^{-n}	n.49	0.32×10^{-n}	n.83	0.15×10^{-n}
n.16	0.69×10^{-n}	n.50	0.32×10^{-n}	n.84	0.14×10^{-n}
n.17	0.68×10^{-n}	n.51	0.31×10^{-n}	n.85	0.14×10^{-n}
n.18	0.66×10^{-n}	n.52	0.30×10^{-n}	n.86	0.14×10^{-n}
n.19	0.65×10^{-n}	n.53	0.30×10^{-n}	n.87	0.13×10^{-n}
n.20	0.63×10^{-n}	n.54	0.29×10^{-n}	n.88	0.13×10^{-n}
n.21	0.62×10^{-n}	n.55	0.28×10^{-n}	n.89	0.13×10^{-n}
n.22	0.60×10^{-n}	n.56	0.28×10^{-n}	n.90	0.13×10^{-n}
n.23	0.59×10^{-n}	n.57	0.27×10^{-n}	n.91	0.12×10^{-n}
n.24	0.58×10^{-n}	n.58	0.26×10^{-n}	n.92	0.12×10^{-n}
n.25	0.56×10^{-n}	n.59	0.26×10^{-n}	n.93	0.12×10^{-n}
n.26	0.55×10^{-n}	n.60	0.25×10^{-n}	n.94	0.12×10^{-n}
n.27	0.54×10^{-n}	n.61	0.25×10^{-n}	n.95	0.11×10^{-n}
n.28	0.53×10^{-n}	n.62	0.24×10^{-n}	n.96	0.11×10^{-n}
n.29	0.51×10^{-n}	n.63	0.23×10^{-n}	n.97	0.11×10^{-n}
n.30	0.50×10^{-n}	n.64	0.23×10^{-n}	n.98	0.11×10^{-n}
n.31	0.49×10^{-n}	n.65	0.22×10^{-n}	n.99	0.10×10^{-n}
n.32	0.48×10^{-n}	n.66	0.22×10^{-n}	n + 1.00	0.10×10^{-n}
n.33	0.47×10^{-n}	n.67	0.21×10^{-n}		

explosive and materials with which it
is likely to come into contact to a pro-
longed, high temperature conditioning,
simulates the possible effect of such
contact under normal conditions.

Apparatus

This test involves the same proce-
dure and apparatus as the vacuum sta-
bility test. The preparation of the spec-
imens for testing may require glass
plates (for film drying) or tools for cut-
ting, rasping, or grinding, as suggested
below.

Preparation

Since the efficiency of the test is
commensurate with the degree of con-
tact between the materials under study,
insure an intimate contact by reducing
all solids to a practicable fineness by
cutting or milling into chips, rasping in-
to shreds or granules, or pulverizing,
observing established safety procedures.

Note: For some of the materials for
which reactivity data may be required,
special preparations will be necessary.
Typical of such materials are the liquid
coating materials. Those which form
removable films (paints, lacquers, etc.)
may be predried on glass plates, stripped,
and chipped. Those which form non-
removable films (metal coatings such as
parkerizing and anodizing) may be de-
posited directly on chips of the metal
involved, and then tested.

Procedure

The procedure for the vacuum stability
test is followed in this test. Normally,
the test temperature is 100°C, but in
special cases the temperature may be
raised to 120°C or lowered to 90°C or
75°C. The duration of the test is 40
hours.

For the basic unit (one explosive and
one contact material) select 3 sample
tubes. Into the first tube place 2.5 ± 0.01
grams of the explosive material, into
the second tube place 2.5 ± 0.01 grams
of the contact material, and into the third
tube place 2.5 ± 0.01 grams of the ex-
plosive material and 2.5 ± 0.01 grams of
the contact material.

Blend the materials which have been
placed in the third tube by appropriate
agitation, being careful not to lose any
of the materials or to get them onto the
ground-glass throat of the sample tube.
(This might make for an insecure junction
between the sample tube and the mano-
meter.) Complete the three assemblies
by joining the manometers to the sample
tubes and proceed as in the vacuum
stability test.

Evaluation

In determining the degree of re-
activity of the materials under test, the
materials processed separately are used
as controls. The reactivity (or chemical

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deterioration) of the explosive is measured by comparing the volume of gas generated by the mixture of the explosive and the chosen contact material with the volume of gas generated by the controls.

The extent of reactivity is then calculated by the following equation:

$$R = C - (A + B)$$

where:

R = extent of reactivity, or volume of gas generated by the mixture in excess of the controls

C = volume of gas generated by the mixture

A = volume of gas generated by the explosive

B = volume of gas generated by the contact material.

Estimate the degree of reactivity by comparing the extent of reactivity (gas volume) with the following adjective-rating table:

Extent of Reactivity, Excess Gas, ml	Degree of Reactivity
Less than 0 (minus value)	None
0 to 1	Negligible
1 to 2	Very slight
2 to 3	Slight
3 to 5	Moderate
5 and over	Excessive

Evaluate the significance of the degree of reactivity as:

Degree of Reactivity	Significance
None to Slight	Essentially non-reactive
Moderate	Possibly deleterious
Excessive	Probably deleterious

ACKNOWLEDGMENT

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